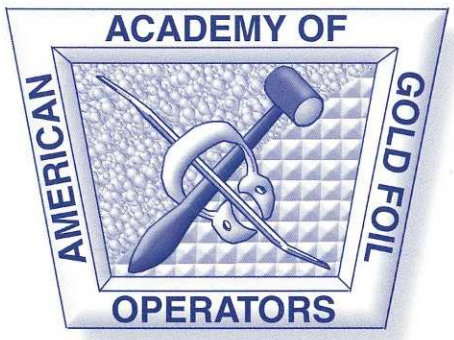
OPERATIVE DENTISTRY



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ACADEMY OF AMERICAN GOLD FOIL OPERATORS

OPERATIVE DENTISTRY

ACADEMY OF OPERATIVE DENTISTRY

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Aim and Scope

Operative Dentistry publishes articles that advance the practice of operative dentistry. The scope of the journal includes conservation and restoration of teeth; the scientific foundation of operative dental therapy; dental materials; dental education; and the social, political, and economic aspects of dental practice. Review papers, book reviews, letters, and classified ads for faculty positions are also published.

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Guest Editorial

Progress and Decline

Operative Dentistry, which succeeded *The Journal of the American Academy of Gold Foil Operators* in 1976, has prospered. Beginning with a quarterly 48-page publication, it is now published bimonthly in a larger 72-page format. The high quality of its articles has been recognized by the Institute for Scientific Information, which for 1999, ranked the impact factor of *Operative Dentistry* as 18th out of 45 international journals in the category of Dentistry, Oral Surgery, and Medicine. Nevertheless, more articles on clinical techniques would be most welcome.

Operative dentistry, as taught in dental schools, however, has declined markedly in quality. Because it comprises, by far, the largest part—about 65 percent of a general practice—operative dentistry should be taught to a high standard. The other branches of dentistry are specialties, and patients can be assured of high-quality treatment. Gold foil, still the best and most durable material for restoring teeth with small lesions, has been almost entirely eliminated from the curriculum. A restoration of gold foil usually lasts for the life of the tooth. It is also likely that the junctional epithelium with hemidesosomes attaches to gold foil, as has been shown to attach to titanium implants. Moreover, the lingual approach to Class III cavities results in an invisible or inconspicuous restoration, so the complaint of the unsightliness of gold foil is not valid here. By removing gold foil from the curriculum, the very institutions charged with scientific leadership are instead promoting mediocrity and denying patients the highest quality of dental treatment. As a result of the decline in the competence of graduates, the standard of examinations for licensure has had to be lowered. For example, a current graduate of the University of Washington would not be able to pass a state board examination, such as that given in the 1950s. With more emphasis on prevention and the decline in the incidence of dental caries, the time spent on operative dentistry should be decreasing. Instead, it is now estimated that 50-70 percent of a dentist's time is required to replace or repair existing restorations (Anusavice, 1995). Additionally, the decline in the competence of students has discouraged skilled operators from joining the faculty.

The administrative bureaucracy of dental schools has grown substantially, and the added expense reduces the funds available for teaching. Committees for selecting deans tend to place great emphasis on higher degrees, research, and publications, so that many deans are specialists and not well versed in the requirements for teaching operative dentistry. Nor do they recognize the importance of laboratory exercises in developing the manual dexterity needed for preparing precise cavities in hard tissue or for carving proper anatomy. As a result, the time allocated to training in the laboratory has been reduced considerably. On two occasions a candidate with excellent skills in operative dentistry, a PhD degree, and demonstrated administrative ability, was rejected. He wanted to address the shortcomings in the teaching of operative dentistry and introduce the study club method of teaching. In one case, the accepted candidate had to be fired for incompetence after two years of a three-year contract. The selection committee, of course, was not held accountable.

Much of the instruction in good operative dentistry now occurs outside dental schools, in study clubs. The Academy of R V Tucker Study Clubs, for example, which concentrates on gold castings, comprises 41 study clubs distributed over North America, Germany, Italy, and Hawaii. Also, a course on gold foil is given annually in Vancouver, Canada—a course that attracts Europeans.

For many years American dental schools were renowned for their teaching of operative dentistry. Dental school graduates in Europe, Australia, and New Zealand would spend a year at an American school to upgrade their skills in operative dentistry and obtain a prestigious DDS degree. A consequence was the formation of an American Dental Society of London and an American Dental Society of Europe. A few years ago the prestigious American automobile industry came under mismanagement and the quality of their cars declined. Fortunately, the Japanese came to the rescue and pro-

vided consumers with cars of high quality. A few years ago the Italians and the Germans were the only ones buying Electromallets. Bad policies, once begun, are difficult to reverse, and it is unlikely in the foreseeable future that American dental schools will make the much-needed changes to revive the quality of the teaching of operative dentistry and graduate dentists who can provide patients with the highest quality of treatment. Under these circumstances, let us help and encourage the Europeans to become the leaders in operative dentistry.

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Commentary

Dr Alexander Ian Hamilton is the founding editor of *Operative Dentistry,* and as such, set the direction, style, and tone of your current journal. He planned and developed the publication for 2 years, and then produced 10 volumes, 40 issues, and 3 supplements during the next 10 years of his tenure. The first use of color in the journal appeared in his final issue in 1985. His editorials have inspired and his enormous contributions have been acknowledged by each of the editors who followed. It would be impossible to calculate the hours of his life (completely unremunerated) that he gladly gave to provide all of us with such an outstanding source of scientific and clinical information.

There is not enough space to even briefly enumerate all of Ian Hamilton's contributions to the dental profession. However, I would refer the interested reader to two previous issues of *Operative Dentistry*: volume 10(4), 1985, which contains a special tribute to Dr Hamilton from his co-workers and volume 11(1), 1986, in which Dave Bales' editorial presents an excellent outline of Dr Hamilton's life.

In my opinion, the letter that accompanied the editorial he was gracious enough to provide for this issue gives the most telling insight into the nature of the man. Not only did he offer encouragement to a fledgling editor, but also politely pointed out a slight discrepancy in the AOD logo (which has been corrected) and informed me of a new *Terminologica Anatomica* that had just been published. He also mentioned that he had been struggling with a biochemistry laboratory course and was now just two courses away from completing his BSc in biochemistry (which will augment his BA, MA in economics, PhD in anatomy, and DDS degrees). It is this ageless dedication, intelligence, and quest for knowledge that delineate the gentleman who provided the solid foundation for this journal.

Dr Hamilton noted in his first Editorial (*Operative Dentistry* 1(1), 1-2, 1976): "A journal needs not only authors and editors but also subscribers and readers—readers with an interest in the success of the journal. If there is a need for *Operative Dentistry*, it shall flourish, but success will depend ultimately on the profession identifying itself with the journal's purpose." It is obvious to me that, in our 25th year of publication, the profession has definitely identified itself with the journal created by Dr Hamilton.

Michael A Cochran
Editor

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Durability of Enamel Bond Strength of Simplified Bonding Systems

M Miyazaki • M Sato • H Onose

Clinical Relevance

Some of the recently introduced simplified bonding systems showed decreased enamel bond strengths after thermal cycling and it was dependent on the systems employed.

SUMMARY

The purpose of this study was to investigate the influence of thermal cycling on enamel bond strength of 2-step bonding systems. The systems used were self-etching primer systems; Imperva Fluoro Bond, Clearfil Liner Bond II, and Mac Bond II; and self-priming adhesive systems; One Step, OptiBond Solo, Prime & Bond 2.0, and Single Bond. Bovine mandibular incisors were mounted in self-curing resin and the facial surfaces were wet ground with #600 SiC paper. Enamel surfaces were treated following each manufacturer's instructions. Adhesives were applied and composites were condensed into a Teflon mold (ø4x2 mm) and light cured. Bonded specimens were divided into four groups; stored in 37°C water for 24 hours, followed by thermal cycling between 5°C and 60°C for 3,000, 10,000, and 30,000 cycles. Ten samples per test group were tested in shear mode at a crosshead speed of 1.0 mm/min. One-way ANOVAs followed by Duncan's multiple range test ($p<0.05$) were done. For self-etching primer systems, a significant decrease in bond strength was observed for the thermal cycling groups. For the self-priming adhesive systems, a small decrease in bond strength was observed but no significant differences were found for thermal cycling groups. The changes in bond strength after thermal cycling were different between the bonding systems.

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INTRODUCTION

Since the introduction of dentin primers (Munksgaard & Asmussen, 1984), three-step bonding systems have increased the acceptance and reliability of resin bonding. These systems require dentin conditioning and priming steps prior to bonding agent application to ensure maximum adhesive strength. In an effort to simplify the bonding procedures, several new two-step adhesive systems have been developed. The systems that use phosphoric acid with an adhesive are called self-priming adhesive systems or one-bottle dental adhesive systems (Swift & others, 1997; Ferrari, Goracci & Garcia-Godoy, 1997). They combine the functions of the dentin primer and bonding agent of three-step systems. On the other hand, self-etching primer systems combine the tooth surface etching and priming steps to simulta-

neously treat enamel and dentin followed by bonding agent application (Barkmeier, Los & Triolo Jr, 1995; Ikemura, Kouro & Endo, 1996; Gordan & others, 1997).

The acid-etch technique has become a standard procedure for surface condition of enamel prior to bonding agent application since the concept of modifying the enamel structure with phosphoric acid was introduced by Buonocore (1955). Retention with higher bond strength of resin composite has been successful by employing this technique with newly-developed clinical techniques. The retentive ability of etched enamel for composite resin is described as a function of the increase in surface area and wettability of the etched enamel (Gwinnett, 1971; Retief, 1973; Silverstone, 1974). The infiltration of bonding resin into the porous zone results in formation of resin tags, establishing micromechanical retention to etched enamel. Self-etching primers form a continuous layer between the composite resin and the tooth surface by simultaneous demineralization with acidic monomers followed by bonding agent penetration into etched enamel. One morphological study of the etched enamel surface demonstrated that the application of the self-etching primer did not result in as deep an enamel etching pattern as did the application of phosphoric acid (Perdigão & others, 1997). Because of the differences in etching patterns of the enamel surface, further studies are required to determine the durability of the enamel bond created by the self-priming adhesive system and the self-etching primer systems.

Table 1. Two-step bonding systems used in this study

Code	System	Conditioner (Main Components)	Lot #	Adhesive (Main Components)	Lot #
FB	Imperva Fluoro Bond	FB Primer (4-AET, HEMA)	A: 079726 B: 079732	FB Bond (4-AET, HEMA, UDMA, Filler)	0797
LB	Clearfil Liner Bond II	LB Primer (5-MNSA, HEMA, Phenyl-P)	A: 038 B: 048	LB Bond (MDP, HEMA, Bis-GMA, Filler)	0056
MB	Mac Bond II	Primer (MAC-10, HEMA, Phosphonated monomer)	A: 005 B: 003	Bonding Agent (MAC-10, Bis-GMA)	004
OS	One Step	Uni Etch (32% Phosphoric acid)	089306	One Step (BPDM, Bis-GMA HEMA, acetone)	059236
OB	OptiBond Solo	Gel Etchant (37.5% Phosphoric acid)	708707	OptiBond Solo (GPDM, HEMA, Bis-GMA, Filler)	710086
PB	Prime & Bond	Conditioner 36 (36% Phosphoric acid)	9604055	Prime & Bond (PENTA, UDMA, R-5-62-1, acetone)	9604181
SB	Single Bond	Etchant (35% Phosphoric acid)	7EC	Single Bond (Bis-GMA, HEMA, Polyalkenoic copolymer)	6AB

4-AET: 4-acryloxyethyltrimellitic acid, HEMA: 2-hydroxyethyl methacrylate, UDMA: urethane di-methacrylate, 5-MNSA: N-methacryloyl 5-aminosalicylic acid, Phenyl-P: 2-methacryloyloxyethyl phenyl hydrogen phosphate, MDP: 10-methacryloyloxydecyl dihydrogen phosphate, Bis-GMA: bisphenol-glycidyl methacrylate, MAC-10: 11-methacryloxy-11-undecarboxylic acid, BPDM: bisphenol di-methacrylate, GPDM: glycerophosphate dimethacrylate, PENTA: phosphoric penta-acrylate ester.

Long-term bonding through micromechanical retention to the etched enamel surface of the cavity preparation is required in order to strengthen the remaining tooth structure. Evaluation for bonding durability is essential since the bond between restoration and tooth substrate is clinically significant only if it is long-lasting (Gwinnett & Wu, 1994; Burrow, Satoh & Tagami, 1996). The purpose of this study was to determine the effect of thermal cycling on the enamel bond strengths of several newly-developed two-step bonding systems to bovine enamel by means of measurement of shear bond strength, fracture mode, and SEM observations of the resin-enamel interface.

METHODS AND MATERIALS

Materials Tested

Three self-etching primer systems; Imperva Fluoro Bond/Lite-Fil II A (FB, Shofu Inc, Kyoto, Japan), Clearfil Liner Bond II/Clearfil AP-X (LB, Kuraray Co, Osaka, Japan), and Mac Bond II/Palfique Estelite (MB, Tokuyama Co, Tokyo, Japan), and four self-priming adhesive systems; One Step/Ælitefil (OS, Bisco Inc, Itasca, IL, 60143), OptiBond Solo/Prodigy (OB, Kerr Corp, Orange, CA, 92867), Prime & Bond 2.0/Spectrum (PB, Dentsply/DeTrey, Konstanz, Germany), and Single Bond/ Z100 (SB, 3M Dental Products, St Paul, MN, 55144) were employed (Table 1). All adhesive systems were used according to the manufacturers' instructions. A curing unit Optilux 401 (Demetron/Kerr, Danbury, CT, 06810) was connected with a variable transformer in order to adjust the light intensity to 600 W/m^2 as measured with a digital radiometer (LI-189, Li-Cor Inc, Lincoln, NE 68504) fitted with a band-pass filter (470 ± 40 nm) on the aperture to limit its response to the most effective wavelengths for curing visible light-cured materials.

Bond Strength Test

Mandibular incisors extracted from two-three year old cattle and stored frozen (-20°C) for up to two weeks were used as a substitute for human teeth. After removing the roots with an Isomet low-speed saw (Buehler Ltd, Lake Bluff, IL, 60044), the pulps were removed and the pulp chamber of each tooth was filled with cotton to avoid penetration of the embedding media. The labial surfaces of bovine incisors were ground on wet 240-grit SiC paper to a flat enamel surface. Each tooth was then mounted in cold-curing acrylic resin to expose the flattened area and placed in tap water to reduce the temperature rise from the exothermic polymerization reaction of the acrylic resin. Final finish was accomplished by grinding on wet 600-grit SiC paper. After ultrasonic cleaning with distilled water for three minutes to remove the excess debris, these surfaces were washed and dried using a three-way syringe.

A Teflon mold, 2.0 mm high and 4.0 mm diameter was used to form and hold the materials to the tooth surface. Resin composite was condensed into the mold and cured for 40 seconds. The Teflon mold and adhesive tape were removed from the specimens 10 minutes after light irradiation. Bonded specimens from each group of materials were divided into four treatment groups of 10 specimens each for testing: Group 1) stored in 37°C water for 24 hours after placement, without thermal cycling, Group 2) stored in 37°C water for 24 hours followed by subjecting to thermal cycling between 5°C and 60°C for 3,000 cycles, Group 3) stored in 37°C water for 24 hours followed by subjecting to thermal cycling for 10,000 cycles, and Group 4) stored in 37°C water for 24 hours followed by subjecting to thermal cycling for 30,000 cycles. The dwell time in each bath was 30 seconds and the transfer time was five seconds.

After treatment the specimens in each group were tested in shear mode using a shear knife-edge testing apparatus in an Instron testing machine (Type 4204, Instron Corp, Canton, MA, 02021) at a crosshead speed of 1.0 mm/minute. Shear bond strength values in MPa were calculated from the peak load at failure divided by the specimen surface area.

After testing, the specimens were examined in an optical microscope at a magnification of x10 to define the location of the bond failure. The test area on the tooth was divided into eight segments and the percentage that was free of material was estimated. The type of failure was determined based on the percentage of substrate free material as: adhesive failure, cohesive failure in resin composite, cohesive failure in bond agent, and cohesive failure in enamel.

The results were analyzed by calculating the mean shear bond strength (MPa) and standard deviation for each group. The data for each group were tested for homogeneity of variance using Bartlett's test, and then subjected to one-way ANOVAs followed by the Duncan multiple range test at $p< 0.05$ to make comparisons among the four treatment groups of each bonding system.

Scanning Electron Microscopy

For the ultrastructure observation of the resin/enamel interface, bonded specimens stored in the same conditions as bond strength test were embedded in epoxy resin, then longitudinally sectioned with the Isomet saw. The sectioned surfaces of the cut halves were polished with diamond pastes. These surfaces were then subjected to Argon-ion beam etching (Type EIS-200ER, Elionix Ltd, Tokyo, Japan) for 30 seconds with the ion beam (accelerating voltage 1.0 kV, ion current density 0.4 mA/cm^2) directed perpendicular to the polished surface. The surfaces were coated in a vacuum evaporator (Quick Coater Type SC-701, Sanyu Denshi Inc, Tokyo, Japan) with a thin film of gold. Observation was done under the scanning electron microscope (JSM-5400, JEOL Ltd, Tokyo, Japan) at an operating magnification of x3500.

RESULTS

The mean shear bond strengths to bovine enamel are shown in Table 2. After 24 hours storage in water, the enamel bond strengths of the self-etching primer systems ranged from 16.4 ± 2.8 ~ 20.0 ± 2.0 MPa, and MB showed significantly higher bond strength than FB and LB. For self-priming adhesive systems, the enamel bond strengths ranged from 19.2 ± 1.5 ~ 24.4 ± 2.3 MPa. No significant differences were found among the systems. The mean enamel bond strengths of all bonding systems decreased with the number of thermal cycles. Significant decreases in enamel bond strengths were observed for self-etching primer systems (FB, LB, and MB), while no differences were found for self-priming adhesive systems (OB, PB, and SB) except OS, which showed an initial decrease in bond strength. No differ-

Table 2. Influence of Thermal Cycling on Bond Strength (Mean ± SD, MPa) of Two-Step Bonding Systems to Bovine Enamel

	Storage Condition			
Code	**24 hours**	**3,000 TC**	**10,000 TC**	**30,000 TC**
FB	17.6 ± 2.1^{a}	13.6 ± 2.7^{b}	13.4 ± 2.3^{b}	9.4 ± 2.5^{c}
LB	16.4 ± 2.8^{d}	11.8 ± 1.8^{e}	11.6 ± 2.1^{e}	8.7 ± 2.1^{f}
MB	20.0 ± 2.0^{g}	16.9 ± 2.4^{h}	16.0 ± 2.8^{h,i}	14.4 ± 1.8^{i}
OS	19.2 ± 1.5^{j}	16.2 ± 1.4^{k}	15.9 ± 2.4^{k}	15.1 ± 2.7^{k}
OB	21.3 ± 1.2^{l}	20.7 ± 2.1^{l}	20.1 ± 1.7^{l}	19.3 ± 1.3^{l}
PB	23.5 ± 2.8^{m}	22.6 ± 2.7^{m}	22.5 ± 1.7^{m}	22.5 ± 1.7^{m}
SB	24.4 ± 2.3^{n}	24.2 ± 2.9^{n}	23.7 ± 2.9^{n}	23.3 ± 2.9^{n}

N=10 TC: Thermal cycling
Rows with the same superscript letters are not significantly different (p<0.05).

ences were found among the thermal cycling groups.

There was a trend toward differences in failure mode among thermal cycling groups. The predominant mode of failure was mixed failures consisting of cohesive in resin and adhesive over the period of thermal cycling for self-priming adhesive systems. For the self-etching primer systems, adhesive failure tended to increase, while cohesive failures tended to decrease with increasing the number of thermal cycles.

The SEM observations of the resin-enamel interface are shown in Figures 1 and 2. After argon ion beam etching, resin tag formation with the infiltration of adhesive resin into the roughened enamel according to the phosphoric acid etching was clearly seen for self-priming adhesive systems. In the 30,000 cycles thermal cycling groups, small cracks were observed at the resin-enamel interface for the self-etching primer systems but were not observed for the self-priming adhesive systems.

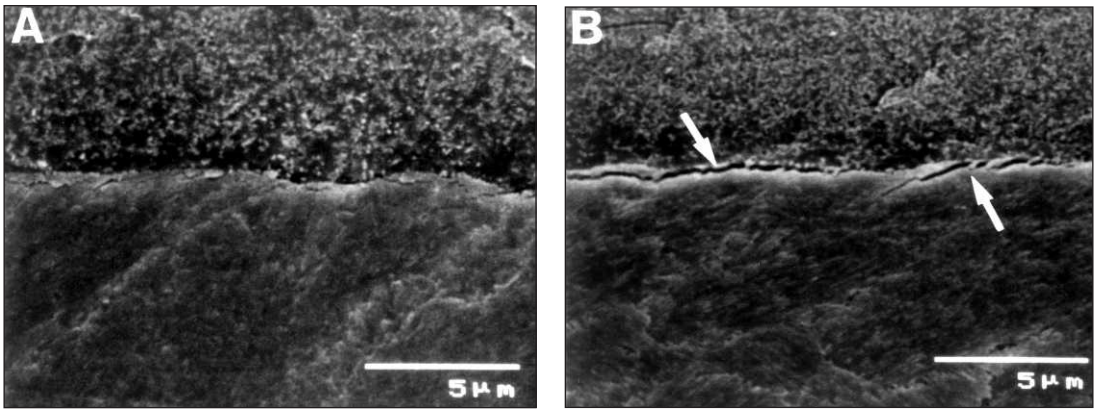

Figure 1. *SEM observations of enamel (E)-resin (R) interface of Clearfil Liner Bond 2 after 24 hours (A) and 30,000 times thermal cycling (B). Small cracks are observed at the interface between the adhesives and etched enamel for 30,000 times thermocycling. (original magnification X3500)*

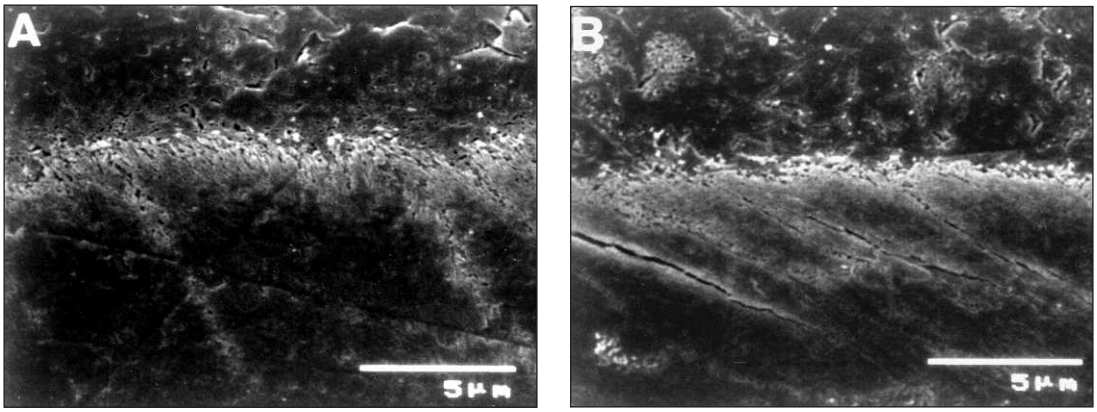

Figure 2. *SEM observations of enamel (E)-resin (R) interface of Single Bond after 24 hours (A) and 30,000 times thermal cycling (B). No gap formation was seen between enamel and adhesives for both treatment groups. (original magnification X3500)*

DISCUSSION

It is difficult to obtain large numbers of intact extracted human teeth for conducting bond strength tests. In this study, bovine teeth were used as a suitable substitute for human teeth, as reported by previous studies (Nakamichi, Iwaku & Fusayama, 1983; Fowler & others, 1992). Most bond strength studies have been done using flattened tooth surfaces of extracted teeth without the presence of pulpal fluid. Care must be taken when drawing conclusions from those studies done under standard laboratory conditions, for it might lead to inappropriate decisions concerning clinical situations (Miyazaki, Oshida & Xirouchaki, 1996).

Although the most reliable conclusions about the performance of dental bonding systems in the oral environment must be derived from long-term clinical trials, long-term storage of the bonded specimen in water or subjecting to thermal cycling may give valuable information about the temperature-dependent degradation of the material. The effect of thermal cycling on dentin bond strength of multi-step bonding systems depends on the bonding systems used and the number of thermal cycles (Burger, Cooley & Garcia-Godoy, 1992; Paul & Scharer, 1993; Davidson, Abdalla & De Gee, 1993). Subjecting bonding specimens to 10,000 thermal cycles indicated that changes in bond strengths after thermal stress depended on the materials used (Hosoya, 1994). This study employed up to 30,000 thermal cycles to accelerate the aging process of the two-step bonding systems.

Water diffusion into the bonding interface between adhesive and tooth surface was found to cause resin to swell and become plasticized (Söderholm, 1991). It has

been reported that the hardness of enamel decreased after storage in physiological saline due to the loss of surface calcium and the softening effect of distilled water was smaller than that of saline (Mühlemann, 1964). Though the degradation in enamel substrate would occur, the rate of degradation and its effect on bond strengths has not been demonstrated.

During the thermal cycling test, the hot water may accelerate hydrolysis of the resin composite as well as the bonding agent and extract poorly polymerized resin oligomers (Bastioli, Romano & Migliaresi 1990). The decreased mechanical properties of resin composite might contribute to the decreased bond strengths for the self-etching primer systems and a self-priming adhesive system (OS). In the shear bond strength test, the stress condition in tooth substrate near the crack tip, where the bond failure initiated, was tensile (Versluis, Tantbirojn & Douglas, 1997). Because the resin tag formation with self-etching primer is relatively shallow and the change in mechanical properties deteriorate after storage in water, the failure tends to occur from the weakened resin tag, which exists between the etched enamel and the resin. The fracture mode of the increased adhesive failure for self-etching primer systems might reveal this.

For the self-etching primer systems, simultaneous etching and priming facilitates penetration of the adhesive resin monomer into etched enamel. The penetration of these acidic monomers into etched enamel creates resin tags. Though the low pH of the self-etching primer allows mineralized tissue to be etched and primed in a single treatment step, this might not be enough to create a stable resin tag strong enough to withstand thermal stress. It has been demonstrated that the solo application of a self-etching primer resulted in a shallow etching pattern which might be the result of deficient penetration of the self-etching primer into the enamel microporosities, or a result of the precipitation of calcium onto the enamel surface masking the etch pattern and interfering with resin penetration (Perdigão & others, 1997). If the resin does not completely infiltrate the etched enamel, a region of unprotected enamel prisms may exist. This region might be susceptible to hydrolytic degradation after long-term storage in water. In the SEM observation of self-etching primer systems (Figure 2), small cracks and porosites were observed between the enamel and adhesive as the number of thermal cycles increased. On the other hand, the self-priming adhesive in the "one-bottle" adhesive systems is a hydrophilic solution that is extremely effective in wetting the etched enamel surface. After the application of phosphoric acid, the application of a self-priming adhesive penetrated the etched enamel and hardened after evaporation of the solvent and light exposure. This process created a stable resin tag and mechanical retention between enamel and resin composite (Figure 1).

Our previous study (Miyazaki & others, 1998) about durability of dentin bond strength of two-step bonding systems suggested that the bond strengths of self-priming adhesive systems decreased with the number of thermal cycles, which may have been caused by the region of demineralized dentin not encapsulated by the adhesive resin. For the self-etching primer systems, the primer might infiltrate the collagen fibers as it decalcifies the inorganic component to the same depth in dentin, so that the small decrease in bond strength was observed after thermal cycling. These results about dentin bond strength were opposite to this study dealing with enamel bond strength. There is some concern that the manufacturers are sacrificing enamel bond strength for the self-etching primer systems, and dentin bond strength for the self-priming adhesive systems in their effort to simplify clinical application. It seems that further research is required to keep up with the demand for convenient bonding procedures with fewer clinical steps, which includes simultaneous treatment of enamel and dentin.

The depth of enamel affected during the etching procedure depends on the type of acid, the acid concentration, the duration of etching, and the chemical composition of the surface enamel (Retief & others, 1986). The question is whether these self-etching primers are capable of providing sufficient etching of enamel such that an adequate micromechanical bond with a bonding agent can be formed. From the results of this study, the benefit of using self-etching primers in terms of simplifying the clinical procedure might be negated by the reduction in bond strength, which was demonstrated by thermal cycle testing. The general practitioner who uses these adhesive systems should understand the factors that influence the durability of the restorations and be aware of their limitations.

CONCLUSIONS

The influence of thermal cycling on enamel bond strengths was different among the bonding systems used. Bond strengths after thermal cycling appear to be more stable for the self-priming adhesive systems than for the self-etching primer systems. The benefits of using some of these bonding systems, in terms of simplifying the clinical procedure, might be negated by the long-term thermal stress.

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Microhardness of Carious Deciduous Dentin

Y Hosoya • SJ Marshall • LG Watanabe • GW Marshall

Clinical Relevance

The dentin in all regions on the carious side of deciduous teeth was significantly softer than sound dentin, and thus, may require different treatments to optimize bonding for restorations.

SUMMARY

The purpose of this study was to measure the microhardness values of deciduous dentin and to compare the values as a function of position in the caries-affected layers of dentin including transparent, adjacent sound dentin, and dentin regions far from and not related to caries. Seven extracted or exfoliated deciduous anterior teeth that had dentin caries on a proximal surface were sectioned parallel to the long axis of the tooth. Ten sectioned and polished specimens were dehydrated and dried. Microhardness was measured with a Knoop indenter and correlated with wet-SEM micrographs. All data were statistically analyzed using a two-way ANOVA with subsequent Tukey-Kramer multiple comparison tests at $p < 0.05$. The hardness decreased from the dentinoenamel junction to the pulp chamber wall, except for the region under the caries. The hardness values of the region under the caries were significantly lower than those of other regions except for the inner region. For both the carious and sound sides, the hardness values of the inner region were significantly lower than those of the outer and middle regions. In comparing the hardness among regions in the carious and sound sides, the hardness of the outer, middle, and inner regions on the carious side was significantly lower than those in the sound side.

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INTRODUCTION

Although permanent-tooth dentin has been studied extensively, the microstructure of dentin in deciduous teeth has received limited attention. A better understanding of dentin in deciduous teeth, especially for carious deciduous dentin, will improve dentin bonding methods and make dental restorations more effective and successful. The hardness of permanent dentin has been studied by a number of researchers (Hodge & McKag, 1933; Totah, 1942; Craig & Peyton, 1958; Craig, Gehring & Peyton, 1959; Nihei, 1959; Nose, 1961; Fusayama, Okuse & Hosoda, 1966; Hegdahl & Hagebo, 1972; Seaman & Shannon, 1979; Ogawa &

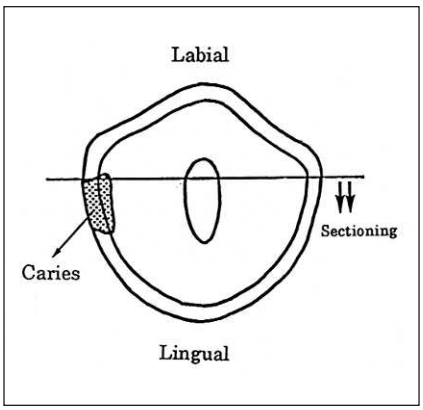

Figure 1A. *Occlusal view of the sectioned specimens. Hardness was measured on the lingual side of the sectioned surface.*

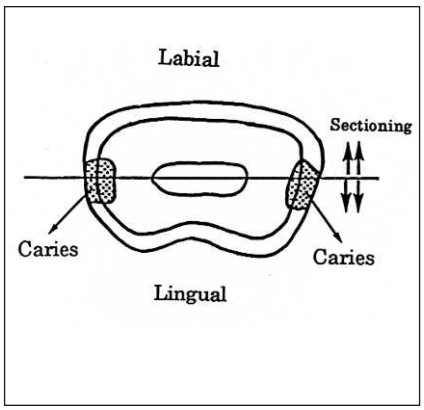

Figure 1B. *Hardness was measured both on the labial and lingual side of sectioned surfaces.*

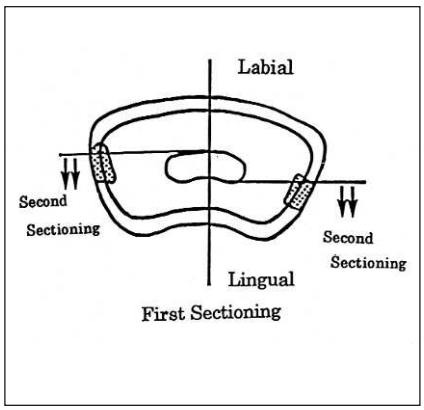

Figure 1C. *Hardness was measured on the lingual side of sectioned surfaces.*

others, 1983; Pashley, Okabe & Parham, 1985; Johnsen, 1994; and Meredith & others, 1996). However, few studies about the hardness of deciduous dentin have been reported (Nihei, 1959; Kume, 1960; Nose, 1961; and Johnsen, 1994). In previous studies, the microcut method (Hodge & McKag, 1933; Totah, 1942), Knoop indenter microhardness test (Craig & Peyton, 1958; Craig & others, 1959; Fusayama & others, 1966; Hegdahl & Hagebo, 1972; Seaman & Shannon, 1979; Ogawa & others, 1983; Pashley & others, 1985; Johnsen, 1994; Meredith & others, 1996), and Vickers indenter method (Nihei, 1959; Kume, 1960; Nose, 1961) were used for measuring hardness of dentin. Previous studies reported that Knoop hardness values for sound permanent dentin ranged from 20 to 83 KHN (Craig & Peyton, 1958; Craig & others, 1959; Fusayama & others, 1966; Seaman & Shannon, 1979; Pashley & others, 1985; Johnsen, 1994; Meredith & others, 1996) and for sound deciduous dentin from 35 to 60 KHN (Johnsen, 1994), depending on the section of the tooth. Fusayama and others (1966) and Ogawa and others (1983) reported the Knoop hardness of carious permanent dentin for the discolored layer, the transparent layer, the subtransparent layer, and sound dentin. Craig and others (1959) also measured the hardness of carious permanent dentin. The hardness of carious deciduous dentin has not been reported.

The purpose of this study was to measure the microhardness values of carious deciduous dentin and to compare the values among regions associated with carious-affected dentin, including transparent dentin and sound dentin. In addition, the microstructural characteristics of these regions were examined using wet-SEM methods.

METHODS AND MATERIALS

Sample Teeth

Seven deciduous anterior teeth (two maxillary canines, two mandibular canines, and three maxillary central incisors) extracted or exfoliated by the eruption of the permanent tooth were used. After sterilization by gamma irradiation (White & others, 1994), the teeth were stored in 4°C physiologic saline solution. All of these teeth had dentin caries on one or both of the proximal surfaces. To identify the carious areas, radiographs were taken using Kodak Dental Film ultra-speed DF-58 (Eastman Kodak Company, Rochester, NY 14650).

Specimen Preparation

The most labially decayed portion of the four teeth that had caries on one of the proximal surfaces was longitudinally sectioned to expose the carious lesion (Figure 1A). Four sectioned specimens and four carious lesions were obtained from these four teeth. For the two out of three teeth that had caries on both proximal surfaces, the teeth were longitudinally sectioned at the central part of the caries (Figure 1B). Four sectioned specimens and eight carious lesions were obtained from these two teeth. One maxillary deciduous central incisor that had caries on both proximal surfaces was first longitudinally sectioned at the center in a mesiodistal orientation of the tooth, and then the most labially decayed portion of each of these specimens was longitudinally sectioned (Figure 1C). Two sectioned specimens and two carious lesions were obtained from this tooth. The total number of sectioned specimens was 10 and of carious lesions was 13. A modified Isomet low-speed saw (Buehler Ltd, Lake Bluff, IL 60044) with a circular diamond blade 0.15 mm thick and copious filtered water were used for the sectioning of the teeth.

Specimens were then polished on wet silicon carbide paper, using grit sizes of 600, 800, and 1200. Final polishing was carried out on felt cloth using 1, 0.3, and 0.05 μm-size aluminum oxide in deionized water. Optical photomicrographs of the polished specimens were taken with a microscope (Carolina Biological Supply Co, Burlington, NC 27215-3387), and the

infected, discolored, transparent, and sound portions of the dentin were observed on color slides.

The specimens were dehydrated with 25, 59, 75, 95, and 100% ethyl alcohol and dried using 100% HMDS (hexamethyldisilazane) to minimize shrinkage due to drying, following the method of Perdigão and others (1996).

A strip of Mylar tape was tacked onto a clean glass microscope slide with the sticky side up. A plastic ring was placed on the tape and the specimen was placed in the center of the ring with polished side down to prevent resin penetration into the dentin. A mixture of COE Tray Plastic cold-cure resin (Coe Laboratories Inc, Chicago, IL 60658) was poured into the ring for embedding the specimens. After cure of the resin, repolishing was done to remove the adhesive materials on the tooth surface.

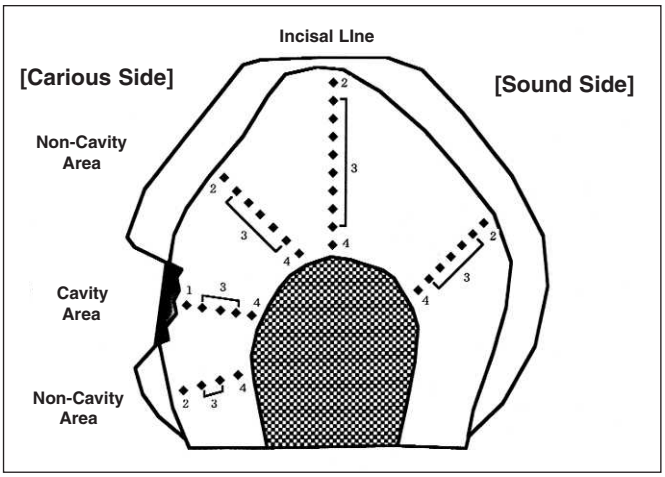

Figure 2. *Names of areas, lines, and regions in microhardness measurement. (Arrow 1: Undercaries Region, Arrows 2 = Outer Region, Bracket 3 = Middle Region, Arrows 4 = Inner Region.)*

Microhardness Preparation

A Miniload 2TM microhardness tester with a Knoop indenter (Ernst Leitz, D-6330 Wetzlar, Germany) using a load of 15 g for 15 seconds was used to produce and measure indentations in specimens. The specimen was mounted on a glass microscope slide with double-sided adhesive tape, and the slide was fixed on the stage on the hardness tester by Mylar tape. Figure 2 shows the names of the areas, line, and regions in the microhardness measurements. Both the carious side and sound side were evaluated by making indentations at intervals of 300 μm from the cementoenamel junction or the incisal edge and perpendicular to the outline of the dentinoenamel junction so that the long diagonal of each indentation was parallel to the dentinoenamel junction. Measurements were made from the dentinoenamel junction to the pulp chamber wall. Under the caries, hardness was determined every 300 μm from the most outer portion of the affected dentin to the pulp chamber wall. Hardness of the infected dentin was not measured because it was too soft. If the indentation was unclear or irregular so that measurements were unreliable, the hardness was determined at the next measuring point. In addition, hardness of the mesiodistally-oriented center portion was determined every 300 μm from the dentinoenamel junction or from the attrited and exposed dentin surface of the incisal edge to the pulp chamber wall. Each of the lines was divided into three regions: the outer region was the first measuring point (300 μm beneath the dentinoenamel junction), the inner region was the innermost measuring point close to the pulp chamber wall, and all of the other measuring points were treated as middle regions. For the lines under the caries, the term "undercaries region" was used instead of the term "outer region."

Table 1: *Microhardness of Carious Deciduous Dentin on Carious Side, Sound Side, and Incisal Line*

Unit: KHN

Region	Carious Side		Sound Side		Incisal Line	
	Number of Measurements	Mean (S.D.)	Mean (S.D.)	Number of Measurements	Mean (S.D.)	Number of Measurements
Undercaries	94	27.6 (9.9)				
Outer	83	44.1 (6.8)	55.4 (17.9)	68	50.1 (30.7)	6
Middle	534	39.3 (6.8)	48.2 (9.8)	146	47.4 (14.9)	25
Inner	55	25.4 (9.6)	30.3 (11.2)	27	43.1 (16.2)	5
Total	766	33.9 (5.4)	48.3 (13.3)	241	48.8 (21.7)	36

* $p<0.05$

SEM Observation

After the microhardness measurement, all specimens were observed using wet BSEM (backscattered scanning microscopy) with a Topcon Modified SX-40A SEM (Topcon Technologies Inc, Paramus, NJ 07652). Infected, discolored, transparent, and sound dentin was observed in the SEM. Infected, discolored, and transparent layers determined by the color slides were marked on tracings of the SEM pictures. All of the positions of the microhardness indentations were plotted on tracings of the SEM pictures to correlate structure-hardness relationships.

Analysis of Microhardness Values

The microhardness values of carious and sound dentin were compared between the measuring lines and regions. All data were statistically analyzed using a two-way ANOVA with subsequent Tukey-Kramer multiple comparison tests at $p<0.05$.

RESULTS

Microhardness Numbers

A total of 1043 microhardness measurements were made on 270 lines.

Table 1 shows the means and standard deviations of the Knoop hardness numbers (KHN) of the specimens. In all of the carious-side, sound-side, and incisal-line measurements, the hardness decreased from the dentinoenamel junction to the pulp chamber wall, except for the undercaries region, which was the softer region of the affected dentin. On the carious side, the hardness of the undercaries region was significantly lower than that of the outer and the middle regions; however, there was no significant difference between the hardness of the undercaries region and that of the inner regions. On both the carious and sound sides, the hardness of the inner region was significantly lower than that of the outer and middle regions, which were not significantly different. The hardness of the outer, middle, and inner regions on the carious side were significantly lower than on the sound side. The overall average hardness on the carious side was significantly lower than that on the sound side. On the line from the incisal, there was no significant difference in hardness.

Table 2 shows the means and standard deviations of the hardness of the undercavity and adjacent noncavity areas on the carious side. In the undercavity area, the hardness of the undercaries region was significantly lower than that of the middle region, but there was no significant difference between the undercaries region and the inner region. The middle region had significantly higher hardness than the inner region. In the adjacent area, the hardness of the inner region was significantly lower than that of the outer and middle regions, which were not significantly different from each other. The hardness of the undercaries region was significantly lower than that of the adjacent outer region in the noncavity area. However, there was no significant difference in hardness in the undercavity area versus the noncavity area for the middle and inner regions or for the overall average values.

SEM Observations

Figures 3A-B and 4A-B are views of selected areas of the specimens after microhardness measurements.

Figure 3A displays the longitudinal cut through a tooth with decay involving both proximal surfaces. Indentation A (Figure 3B), indented on the transition zone between the carious dark region and transparent dentin, was bigger than indentations at B and C in the transparent and in the sound dentin below the transparent dentin, respectively. Thus, hardness was lower at A than B or C. The hardness of area B was lower than that of area C.

Figure 4A shows the longitudinal cut through the carious dark region on one interproximal surface. The region of A (Figure 4B) illustrates the discolored region. The dentin matrix around the tubules was demineralized, and amorphous material was observed in some of the dentinal tubules. In region B, dentinal tubules were widely opened and an early stage of partial occlusion of the tubules was observed. In region C, the diameters of the tubules were smaller than those in region B. In some of the specimens, the tubules in the transparent zone were partially

Table 2: *Microhardness of Carious Deciduous Dentin on Carious Side*

Unit: KHN

Region	Under Cavity Area		Non-Cavity Area	
	Number of Measurements	Mean (S.D.)	Mean (S.D.)	Number of Measurements
Undercaries	94	27.6 (9.9)		
Outer			43.7 (7.3)	83
Middle	304	38.0 (5.4)	40.50 (9.8)	230
Inner	28	23.2 (11.9)	26.6 (13.1)	27
Total	426	30.7 (5.6)	36.9 (8.8)	340

* $p<0.05$

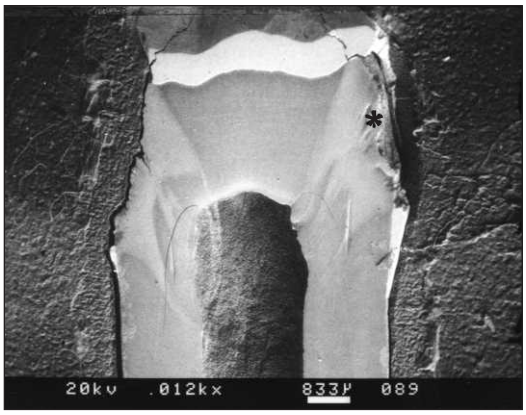

Figure 3A. *Mesial and distal proximal surfaces decayed in dentin caries.*

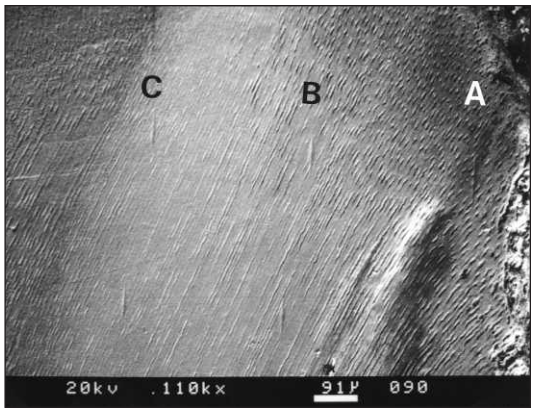

Figure 3B. *Higher magnified views of * area in Figure 3A. Indentation A, indented on the transition zone between the carious dark region and transparent dentin, was bigger than those of B and C and the hardness was lower at A than B or C.*

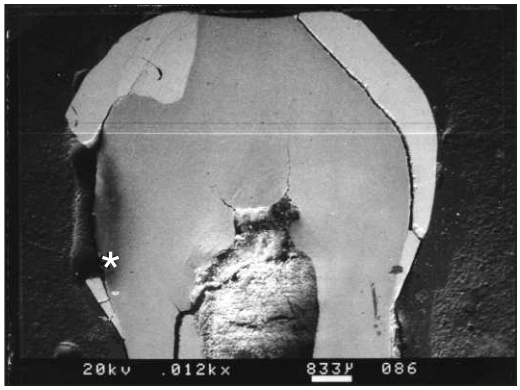

Figure 4A. *Mesial proximal surface decayed in dentin caries.*

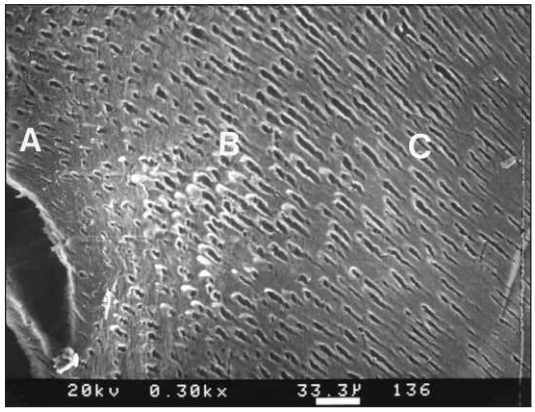

Figure 4B. *Higher magnified view of * area in Figure 4A. The region of A was the discolored region and the dentin matrix around the tubules was demineralized. In region B, dentinal tubules were widely opened, and in region C, the diameter of the tubules was smaller than those in region B.*

occluded. In some of the specimens, the tubules of the adjacent sound dentin were occluded with cylindrical or crystal-like materials. This suggested that what appears to be sound dentin may be a thin area of transparent dentin.

Generally, in the area in which the ratio of intertubular dentin to tubule area was high and the diameter of the tubules was small, the hardness was high. On the other hand, in the area of low hardness, one of which was located beneath the caries, the diameter of the tubules was large and the ratio of intertubular dentin to tubule area was low. In another region close to the pulp, which had low hardness values, the tubules were more numerous and had larger diameters than in other regions.

Figure 5 shows representative hardness values corresponding to the specimen in Figure 4A. The hardness decreased both in the carious and sound sides with distance from the dentinoenamel junction to the pulp chamber wall, and also decreased along the incisal line except for the undercaries region on the carious side. The hardness of the transparent layer was variable.

Transparent dentin could be identified in 12 of 13 carious lesions and generally had reduced hardness. In nine of 12 specimens, transparent dentin was softer than sound dentin. Thus, it appears that transparent dentin in deciduous teeth is generally soft rather than sclerotic.

DISCUSSION

In this study, the Knoop hardness values for the sound side of carious deciduous dentin (Table 1) ranged from 30 to 55 KHN (average: 48 KHN), in good agreement with the previous study (Johnsen, 1994) of deciduous dentin, except in the middle region. The Knoop hardness value of the middle region in this study was higher than that previously reported (Johnsen, 1994).

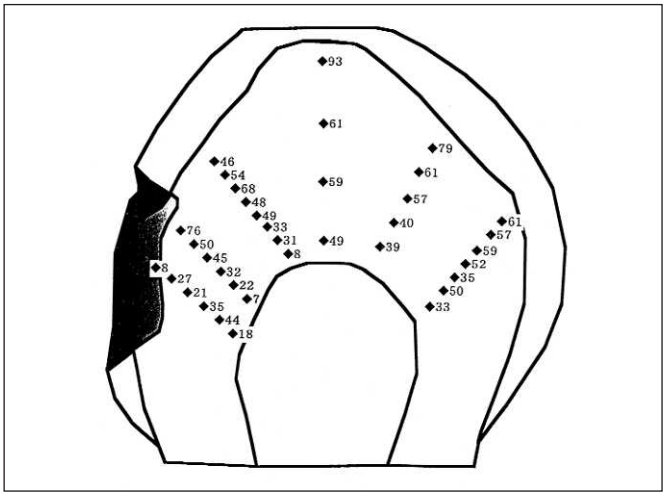

Figure 5. *Representative Knoop hardness numbers for Figure 4A.*

Some studies have reported that in both deciduous and permanent sound dentin, the hardness of peripheral dentin was lower than that of central dentin, and the hardness of the pulpal dentin was the lowest (Craig & others, 1959; Nihei, 1959; Johnsen, 1994). Lower hardness values 100 to 200 μm from the dentinoenamel junction were also reported (Craig & Peyton, 1958; Nihei, 1959), probably due to the presence of mantle dentin. Meredith and others (1996) reported that the hardness of dentin decreased with distance from the dentinoenamel junction. Pashley and others (1985) reported that there was a highly statistically significant inverse correlation between dentin microhardness and tubule density.

In this study the inner region of sound dentin had the lowest hardness, which was significantly lower than the hardness of the outer and middle regions. The hardness of the outer region appeared higher than the hardness of the middle region, but there was no significant difference (Table 1). Since the number of measured points and lines varied among specimens, all points were treated as coming from the middle region except for the two extreme points, one 300 μm from the dentinoenamel junction (outer region) and the other, the innermost point (inner region). Thus, the range of the middle region was large in this study, and the outer region did not consist of mantle dentin.

In the previous studies, sample teeth used for hardness measurement were fixed or stored in a formalin solution (Totah, 1942; Fusayama & others, 1966; Hegdahl & Hagebo, 1972; Ogawa & others, 1983), physiologic saline solution (Kume, 1960; Nose, 1961), deionized water (Meredith & others, 1996), phosphate buffered saline (Pashley & others, 1985), or distilled water (Nihei, 1959), and generally neither dehydration nor drying was attempted on the sectioned specimens. Microhardness is influenced by the loading weight and dryness of the specimens. Hegdahl and Hagebo (1972) measured the load dependence of the Vickers microindentation hardness measurement and found that with increased load there was increased hardness of enamel but a decrease in hardness of dentin. A significant, but slight variation in hardness was obtained when the load was varied within the range of practical applicability, such as the 15 g load we used. The length of the longer diagonal of the Knoop hardness indentations could be accurately measured in our study except for the undercaries region in the undercavity area. In this region, the hardness was very low and the length of the longer diagonal of some indentations was so large that a 10 g load might have been more useful for indenting these areas. However, for indenting the sound deciduous dentin, the 15 g load gave the best results.

Totah (1942) used the microcut method and found that dentin hardness increased 42% by drying, while Nose (1961) reported that with the Vickers microindentation method hardness slightly increased by drying. However, there are no known reports concerning dentin hardness differences between fixed and nonfixed specimens using more modern hardness measurement methods. The influence on the hardness of fixing and drying dentin is still unclear. In this study, the specimens were dehydrated using ethyl alcohol graded solutions and dried by 100% HMDS for one hour to minimize shrinkage and disruption of the structure due to drying stresses. Then the specimens were stored in a desiccator. Thus, the data presented were for completely dried deciduous dentin hardness that had not been subjected to air drying, which is known to result in significant shrinkage of partially demineralized dentin. Further study should be done to clarify the influence of this dehydration and drying method to the dentin hardness.

Nose (1961) and Johnsen (1994) compared the hardness of deciduous and permanent dentin and found that deciduous dentin was slightly softer than permanent dentin. Peripheral and circumpulpal areas were similar in hardness in both dentitions, but the central area of coronal dentin was considerably harder in permanent teeth than in primary teeth (Johnsen, 1994). Comparison of the hardness of the sound deciduous dentin in this study with previously reported sound permanent dentin (Craig & Peyton, 1958; Craig & others, 1959; Fusayama & others, 1966; Seaman & Shannon, 1979; Pashley & others, 1985; Johnsen, 1994; Meredith & others, 1996) showed that the hardness of the outer region in this study was similar to that of permanent dentin. However, the hardness of middle and inner regions in this study was lower than in the previous reports (Craig & Peyton, 1958; Craig & others, 1959; Fusayama & others, 1966; Seaman & Shannon, 1979; Pashley & others, 1985; Johnsen, 1994; Meredith &

others, 1996). Hardness is believed to be related to the degree of mineralization. Thus, it can be speculated that permanent dentin is more highly mineralized.

Underincisal and undercuspal dentin hardness is probably influenced by attrition. Mendis and Darling (1979a) reported that with attrition, there was evidence of progressive formation of intratubular (peritubular) dentin, an increasing number of closed dentinal tubules, and a reduction in the number of open tubules from the pulp side toward the worn surface. Mendis and Darling (1979b) also reported that in longitudinal fractures of attrition, the tubules at the worn surface were completely occluded. Below the zone of closure, the tubules at the worn surface were completely occluded with much of the tubule lumen filled with various-sized crystals. In this study, there was no significant difference in hardness among the regions in the incisal line (Table 1). However, large standard deviations were obtained in all of the regions in the incisal line in our study, probably because the dentin was exposed by attrition in some specimens in which the tubules were mostly closed, but covered with attrited enamel in other specimens in which many tubules were open. Additional variation might depend on the degree of mineralization that might differ with the type of tooth, and there may also be tooth-to-tooth hardness differences.

The hardness of carious deciduous dentin has not been reported. Craig and others (1959) reported that permanent dentin surrounding a caries lesion had a hardness 10 KHN greater than normal dentin, whereas the hardness values at the center of the lesion were much lower, and transparent dentin was 10 KHN harder than the adjacent area. Fusayama and others (1966) and Ogawa and others (1983) measured the hardness of carious permanent dentin and reported that the Knoop hardness for the discolored layer ranged from 20 to 27 KHN, for the transparent layer from 27 to 48 KHN, for the subtransparent layer from 48 to 68 KHN, and for sound dentin from 21 to 68 KHN. In this study, the average Knoop hardness values (standard deviations) of the deciduous dentin on the carious side in the regions under the caries, the middle region, and inner region were 27.6 (9.9), 38.0 (5.4), and 23.2 (11.9) KHN, respectively.

Fusayama and others (1966) stated that steep peaks in hardness curves were often observed immediately before the softening front because of the sclerosed dentin. However, Ogawa and others (1983) reported that the transparent layer was not sclerotic, and this layer was much softer than was normal dentin. In this study, the transparent layer was observed in the middle region on the carious side. The hardness of this region was significantly higher than that of the more superficial or inner underdecay layers, but there was no significant difference between the hardness of the middle regions directly under or to the side of the decay (Table 2). These areas may have involved the formation of transparent dentin. However, the hardness of the middle region in the undercavity area was significantly lower than that of the sound side (Tables 1 and 2). These results were in agreement with the results reported for permanent dentin by Ogawa and others (1983). It can be seen from Figure 5 that the hardness of the transparent layer was variable. It is supposed that the microhardness of the transparent layer is influenced by the degree of remineralization, the depth of the decay, and the distance from the dentinoenamel junction. The hardness of the transparent layer in the outer region might be expected to be higher than that in the inner region. Shorter-interval hardness measurements should be required to justify the relationship between the hardness of the carious lesion and the depth of the dentin.

In the undercaries region (Figure 4B), the dentin matrix was demineralized as a result of the caries. Marshall and others (1989) observed carious permanent dentin by backscattered scanning electron microscopy using the same method employed here. They observed the same number of partially occluded tubules in the transparent zone as in this study (Figure 4B) but less mineralized and clefted tubules in the surrounding dentin. In this study, the diameter of the tubules and the ratio of intertubular dentin to tubule area influenced the hardness. The relationship between the occluded tubule area and the hardness should be clarified in further study. Fusayama and others (1966) reported that the hardness of the discolored layer differed between the acute cases and the chronic cases. The hardness of decayed deciduous dentin might vary with the type and depth of decay, but we were unable to make a clear classification of acute or chronic caries in this study.

In this study, in the comparisons of the carious and the sound sides, the hardness of the outer, middle, and inner regions on the carious side were significantly lower than those on the sound side (Table 1). This result shows that not only in the undercaries region but also in all of the other regions, the hardness of the carious side is significantly lower than that of the sound side. The significantly lower hardness of the carious side might have a deleterious effect on resin adhesion to the dentin. After etching, the hardness might be even lower, so that bonding to these areas might require specific etching treatments that have not yet been defined. Inagaki and others (1989) reported that high correlation has been obtained between the pH of a primer and dentin hardness as well as between hardness of treated dentin and the wall-to-wall polymerization contraction of a light-curing microfilled resin. Van Meerbeek and others (1993) reported that the hardness

of the resin-dentin interdiffusion zone was significantly lower than that of unaltered dentin. They also reported that a gradient in modulus of elasticity was observed from the rather stiff dentin over a more elastic resin-dentin interdiffusion zone and adhesive resin layer to the restorative composite.

Nakajima and others (1995) used a microtensile bond strength test to compare bond strengths made to either caries-affected dentin or normal dentin, using three commercial adhesive systems and indicated that the strength of adhesion to dentin depends upon both the adhesive system used and the type of dentin. Hosoya and others (1996, 1997) compared resin adhesion to deciduous and to permanent bovine dentin and reported that the percentages of cohesive resin fracture for deciduous dentin were lower than those for permanent dentin. However, the shear bond strengths to deciduous dentin were significantly higher than those to permanent dentin. It is supposed that the elasticity of deciduous bovine dentin is higher than that of permanent bovine dentin, and the elasticity difference contributes to the different bond strength and fracture modes between dentin and resin. Further study is required for understanding the precise mechanism of adhesion between carious dentin and resinous materials.

CONCLUSIONS

1. In sound deciduous dentin from anterior teeth, microhardness decreased from the dentinoenamel junction to the pulp chamber wall.

2. The microhardness of the undercaries region and the hardness of the innermost region both on the carious and sound sides was significantly lower than those of other regions.

3. In comparing the hardness between regions on the carious and sound sides, the hardness of all regions on the carious side was significantly lower than on the sound side, suggesting that they were affected by the caries.

4. Transparent dentin could be identified in 12 of 13 lesions and had reduced hardness. In nine of 12 transparent regions, the transparent dentin was softer than sound dentin. Thus it appears that transparent dentin in deciduous teeth is generally soft, rather than sclerotic.

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Single-Crystalline Ceramic Whisker-Reinforced Carboxylic Acid-Resin Composites with Fluoride Release

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Clinical Relevance

Carboxylic acid-resin composites containing ceramic whiskers and fluorosilicate glass release fluoride and possess flexural strength and work-of-fracture substantially higher than those of a traditional glass ionomer, a resin-modified glass ionomer, or a compomer.

SUMMARY

Currently available glass-ionomer, resin-modified glass-ionomer, and compomer materials have relatively low strength and toughness and, therefore, are inadequate for use in large stress-bearing posterior restorations. In the present study, ceramic single-crystalline whiskers were mixed with fluorosilicate glass particles and used as fillers to reinforce experimental carboxylic acid-resin composites. The carboxylic acid was a monofunctional methacryloxyethyl phthalate (MEP). Five mass fractions of whisker/(whisker + fluorosilicate glass), and corresponding resin/(resin + MEP), were evaluated. Four control materials were also tested for comparison: a glass ionomer, a resin-modified glass ionomer, a compomer, and a hybrid composite resin. Flexural specimens were fabricated to measure the flexural strength, elastic modulus, and work-of-fracture (an indication of toughness). Fluoride release was measured by using a fluoride ion selective electrode. The properties of whisker composites depended on the whisker/(whisker + fluorosilicate glass) mass fraction. At a mass fraction of 0.8, the whisker composite had a flexural strength in MPa (mean ± sd; n = 6) of 150 ± 16, significantly higher than that of a glass ionomer (15 ± 7) or a compomer control (89 ± 18) (Tukey's multiple comparison test; family confidence coefficient = 0.95). Depending on the ratio of whisker:fluorosilicate glass, the whisker composites had a cumulative fluoride release up to 60% of that of a traditional glass ionomer. To conclude, combining ceramic whiskers and fluorosilicate glass in a carboxylic acid-resin matrix can result in fluoride-releasing composites with significantly improved mechanical properties.

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INTRODUCTION

It had been reported that approximately half of all restorative dentistry involved the replacement of existing restorations, and about 40% of all replacements were attributed to recurrent caries (MacInnis, Ismail & Brogan, 1991). There is strong evidence that the sustained release of small amounts of fluoride could be a

substantial benefit for a dental restoration because the fluoride could enrich neighboring enamel or dentin to combat secondary caries (Wesenberg & Hals, 1980; Palenik & others, 1992; Leinfelder, 1993; Benelli & others, 1993; Souto & Donly, 1994; Hsu & others, 1998). Glass ionomer (Wilson & Kent, 1972; Prosser, Powis & Wilson, 1986; McLean, 1990) and resin-modified glass-ionomer materials (Mathis & Ferracane, 1989; Mitra, 1989) have received much attention due to their significant release of fluoride, the uptake of fluoride into cavity walls and plaque, and the enhanced reprecipitation of calcium and phosphate promoted by the fluoride release (Wesenberg & Hals, 1980; Swartz, Phillips & Clark, 1984; Swift, 1989a, b; Mitra, 1991; Hicks & Flaitz, 1992; Benelli & others, 1993; Díaz-Arnold & others, 1995). The inferior mechanical properties of glass-ionomer and resin-modified glass-ionomer materials, however, have limited their use (Prosser & others, 1986; Lloyd & Butchart, 1990; Nicholson, Anstice & McLean, 1992; Mitra & Kedrowski, 1994; Kao, Culbertson & Xie, 1996). It was predicted that "the most intractable problem is likely to be lack of strength and toughness" (Wilson & McLean, 1988). The addition of a resin in the matrix did not significantly reduce the problems of glass-ionomer materials (Sidhu, Sherriff & Watson, 1997).

Recently, a novel method of ceramic whisker reinforcement was developed that resulted in dental composite resins with substantially increased strength and toughness (Xu & others, 1999a). Currently available composite resins, although significantly improved (Willems & others, 1992; Anusavice, 1996; Bayne & Thompson, 1996; Ferracane & others, 1997; Eick, Kaufman & Chappelow, 1997; Loza-Herrero & others, 1998), are still inadequate for use as large stress-bearing posterior restorations involving cusps (Wilder, Bayne & Heymann, 1996; Anusavice, 1996). In the ceramic whisker reinforcement method (Xu & others, 1999a), silica glass particles were fused onto high-strength, fine-sized ceramic single-crystalline whiskers at high temperatures. These whiskers were then silanized and incorporated into dental resins. A nearly twofold increase was achieved in the flexural strength and fracture toughness of this improved composite resin. In addition, the composite's resistance to contact damage and microcracking was also improved. The whisker composite restoration had a whitish color and a surface roughness after clinical polishing similar to that of a conventional composite resin filled with fine glass particles (Xu & others, 1999b). However, these studies did not investigate the ceramic whisker reinforcement of dental restorative materials that release fluoride.

The aim of the present study was to investigate the ceramic whisker reinforcement of dental restorative materials with fluoride release. Fluorosilicate glass particles were mixed with ceramic whiskers and used as a filler for a dental resin containing a carboxylic acid monomer. Five whisker/(whisker + fluorosilicate glass) mass fractions of 0.5, 0.7, 0.8, 0.9, and 1.0 were evaluated. The resin/(resin + carboxylic acid monomer) mass fraction was equal to the whisker/(whisker + fluorosilicate glass) mass fraction for each composite. The whisker composites were tested for flexural strength, elastic modulus, work-of-fracture, and fluoride release. Four control materials representative of acceptable clinical performance and different levels of fluoride release were also tested: a traditional glass ionomer, a resin-modified glass ionomer, a compomer, and a hybrid composite resin.

METHODS AND MATERIALS

Filler Powder Preparation

Ceramic silicon nitride single-crystalline (b-Si3N4) whiskers (UBE Industries, Ltd, Tokyo, Japan) with diameters ranging from 0.1 μm to 1.5 μm (mean = 0.4 μm) and lengths ranging from 2 μm to 20 μm (mean = 5 μm) were mixed with fumed silica having a nominal particle size of 0.04 μm (Aerosil OX50; Degussa Corp, South Plainfield, NJ 07080). A whisker:silica mass ratio of 2:1 was used, and the mixture was dispersed by stirring in ethyl alcohol with a magnetic stir bar under vacuum until dry. The silica particles were fused onto the surfaces of whiskers to facilitate silanization and to roughen the whisker surface for enhanced retention in the matrix. To fuse the silica particles onto the whiskers, the dried mixture was heated in air for 30 minutes at a temperature of 800°C. The heat-treated powder was silanized by mixing it with 0.04 mass fraction of 3-methacryloxypropyltrimethoxysilane (MPTMS) and 0.02 mass fraction of n-propylamine in cyclohexane using a rotary evaporator in a 90°C water bath until dry (Xu & others, 1999a).

The silanized whiskers were manually mixed by use of spatulation with an ion-leachable fluorosilicate glass (strontium aluminofluorosilicate; Caulk/Dentsply, Inc, Milford, DE 19963) of particles with sizes ranging approximately from 0.5 μm to 10 μm with a mean of about 2 μm. The fluorosilicate particles were deliberately not silanized to promote their reaction with the carboxylic acid monomer of the resin system and allow for maximum fluoride release. The following whisker:fluorosilicate glass mass ratios were evaluated: 1:1, 7:3, 8:2, 9:1, and 1:0. These ratios corresponded to a whisker/(whisker + fluorosilicate) mass fraction of 0.5, 0.7, 0.8, 0.9, and 1.0, respectively. Whisker mass fractions less than 0.5 were not tested, as the amount of whiskers may not be sufficient for substantial improvement in the composite mechanical properties.

Resin-Carboxylic Acid Monomer Preparation

Modification of a two-part, chemically-activated resin was used to prepare the experimental carboxylic acid-

resin systems. Part I, the initiator resin, consisted of monomers in mass fractions of 0.48975 BIS-GMA (bisphenol glycidyl methacrylate) and 0.48975 TEGDMA (triethylene glycol dimethacrylate), with 0.0005 2,6-di-tert-butyl-4-methylphenol (BHT) and 0.02 benzoyl peroxide (BPO). Part II, the accelerator resin, consisted of mass fractions of 0.495 BIS-GMA and 0.495 TEGDMA, with 0.01 N,N-dihydroxyethyl-p-toluidine (DHEPT) as the polymerization accelerator. A monofunctional carboxylic acid monomer (methacryloxyethyl phthalate, or MEP) (Lot 430-49; Esschem Inc, Linwood, PA 19061) was added to each of the resins to form MEP-resin two-part systems. No additional BPO or DHEPT was added to the resins. Five MEP-resin systems were made with the following resin/(resin + MEP) mass fractions: 0.5, 0.7, 0.8, 0.9, and 1.0.

Paste Preparation and Specimen Fabrication

The five different resin/(resin + MEP) mass fractions were designed to be the same as the five whisker/(whisker + fluorosilicate glass) mass fractions. In mixing the filler powder with the MEP-resin liquid to form a paste, the mass fraction of whisker/(whisker + fluorosilicate glass) was equal to that of resin/(resin + MEP). For example, the liquid with a resin/(resin + MEP) mass fraction of 0.5 was only mixed with the filler powder with a whisker/(whisker + fluorosilicate glass) mass fraction of 0.5. For each mass fraction, two pastes were mixed manually by use of spatulation: the initiator MEP-resin and whisker-fluorosilicate paste, and the accelerator MEP-resin and whisker-fluorosilicate paste. A filler level of a mass fraction of 55% was used for each paste (Xu & others, 1999a). Equal masses of the two pastes were then mixed by spatulation, filled into a mold and hardened in approximately two minutes to make a specimen. The flexural specimens had dimensions of approximately 2 mm x 2 mm x 20 mm. The specimens for fluoride release testing had dimensions of approximately 2 mm x 2 mm x 8 mm. Each specimen was incubated at 37°C for 15 minutes to cure chemically, and then demolded. A total of 60 flexural specimens were made for the whisker composites, with 12 specimens at each whisker/(whisker + fluorosilicate glass) mass fraction. A total of 20 fluoride release specimens were made with four specimens at each whisker/(whisker + fluorosilicate glass) mass fraction.

Specimens of four control materials were also fabricated to provide representative reference for mechanical properties and fluoride release. The first control was a traditional glass ionomer (Ketac-Bond; ESPE, Seefeld, Germany). The manufacturer has recommended a powder:liquid weight ratio of 3.4:1 for bonding applications and a higher ratio of 4-5:1 for crown post build-ups. In the present study, a powder:liquid weight ratio of 5:1 was used and the paste was filled into steel molds and hardened to make specimens. A resin-modified glass ionomer (Vitremer; 3M Dental Products, St Paul, MN 55144) was mixed to form a paste according to the manufacturer's instructions, filled into the mold, and light cured (Triad 2000; Dentsply International, Inc, York, PA 17405) for one minute on each side of the specimen. A compomer (Dyract; Caulk/Dentsply) was filled into the mold and light cured for one minute on each side of the specimen. Specimens of the traditional glass ionomer, the resin-modified glass ionomer, and the compomer were kept in the molds covered with Mylar strips plus glass slides that then were mechanically clamped. They were incubated in a humidor to minimize dehydration at 37°C for 24 hours and then demolded and tested as described below. A hybrid composite resin (TPH; Caulk/Dentsply), consisting of silicate and barium glass particles of about 0.8 μm in diameter and a filler level of 78% mass fraction in a matrix resin of TEGDMA plus a urethane-modified BIS-GMA, was light cured for one minute on each side of the specimen. The TPH specimens were incubated at 37°C for 24 hours and then demolded. The dimensions of the control specimens were the same as those of the whisker composite specimens. A total of 48 flexural specimens were made with 12 specimens for each control material. A total of 16 fluoride release specimens were made with four specimens for each control material.

Mechanical Testing and Fluoride Release Measurement

The flexural specimens were treated in two groups to evaluate the effect of water immersion on composite properties. Each group had 54 specimens with six specimens for each of the nine materials: five whisker composites and four control materials. For the first group, the flexural specimens were incubated and demolded as described above. They were then, without any immersion in water, fractured in a flexural test. The glass-ionomer and resin-modified glass-ionomer specimens were fractured within one hour after being taken out of the humidor to minimize dehydration. For the second group, the flexural specimens were demolded and additionally immersed in distilled water at 37°C for 24 hours prior to the flexural test.

A standard three-point flexural test (American Society for Testing and Materials, 1984) with a span of 10 mm was used to fracture the specimens at a crosshead speed of 0.5 mm per minute on a computer-controlled Universal Testing Machine (model 5500R; Instron Corp, Canton, MA 02021). The following properties were evaluated: flexural strength, elastic modulus, and work-of-fracture (the energy required to fracture the specimen obtained from the area under the load-displacement curve normalized by the specimen's cross-sectional area).

Table 1a: *Mechanical Properties of Specimens Without Water Immersion (mean* ± SD; n=6)*

Whisker/[whisker + fluorosilicate glass] (mass fraction)	Flexural strength (MPa)	Elastic modulus (GPa)	Work-of-fracture (kJ/m^2)
0.5	121 ± 2	7.2 ± 0.2	1.2 ± 0.1
0.7	154 ± 11	8.2 ± 0.4	1.8 ± 0.3
0.8	159 ± 14	8.0 ± 0.7	2.1 ± 0.4
0.9	178 ± 16	7.6 ± 0.9	3.5 ± 0.4
1.0	195 ± 8	7.1 ± 0.5	3.9 ± 0.5
control: traditional glass ionomer	54 ± 6	12.2 ± 1.6	0.16 ± 0.03
control: resin-modified glass ionomer	60 ± 6	5.8 ± 0.7	0.4 ± 0.1
control: compomer	120 ± 9	6.5 ± 0.7	1.5 ± 0.3
control: hybid composite resin	134 ± 18	7.3 ± 0.7	1.7 ± 0.6

*Flexural strength is the highest at whisker/[whisker + fluorosilicate glass] of 1.0, followed by those at 0.9 and 0.8, and the lowest for glass ionomer and resin-modified glass ionomer (Tukey's multiple comparison test; family confidence coefficient - 0.95).

Table 1b: *Mechanical Properties of Specimens After 24 h Immersion in Distilled Water at 37° C**

Whisker/[whisker + fluorosilicate glass] (mass fraction)	Flexural strength (MPa)	Elastic modulus (GPa)	Work-of-fracture (kJ/m^2)
0.5	121 ± 5	5.4 ± 0.9	1.8 ± 0.2
0.7	140 ± 6	6.1 ± 0.2	2.2 ± 0.3
0.8	150 ± 16	7.4 ± 0.3	2.1 ± 0.5
0.9	180 ± 12	6.8 ± 0.9	3.3 ± 0.2
1.0	196 ± 10	7.2 ± 0.4	4.0 ± 0.7
control: traditional glass ionomer	15 ± 7	5.1 ± 2.4	0.04 ± 0.02
control: resin-modified glass ionomer	39 ± 6	5.3 ± 0.2	0.2 ± 0.1
control: compomer	89 ± 18	6.1 ± 0.3	1.8 ± 0.3
control: hybid composite resin	120 ± 16	4.9 ± 0.8	2.4 ± 0.8

*Strength is the highest at whisker/[whisker + flurosilicate glass] of 1.0 and 0.9 followed by those at 0.8 and 0.7, lower for hybrid composite and compomer, and the lowest for glass ionomer (Tukey's multiple comparison test; family confidence coefficient = 0.95).

Fluoride release was measured after storage periods of 1, 7, 14, 30, 60, and 90 days. To minimize dehydration and surface cracking (Mathis & Ferracane, 1989), the glass-ionomer specimens were immersed in distilled water within one hour after they were taken out of the humidor and demolded. Four bar specimens were tested for each material. Each bar was immersed in 2.5 mL water in a capped polystyrene tube (Falcon 2054; Becton Dickinson, Franklin Lakes, NJ 07417) stored in a 37°C oven. Preliminary studies indicated that 2.5 mL of water was sufficient for one Ketac specimen to avoid fluoride saturation of the solution. The amount of fluoride release of some of the whisker composites with high whisker contents was relatively low, and a large amount of storage water (eg, 10 mL) resulted in a low fluoride concentration and hence lower measurement accuracy. After each prescribed storage period, 1 mL of the storage solution was collected from each tube; 1 mL fresh deionized water was then added to the tube for further storage. A buffer solution of 1 mL (TISAB II; Orion Research, Inc, Boston MA 02129) was added to the collected solution for fluoride concentration measurement. A fluoride selective electrode (model 94-09; Orion Research, Inc) was used to measure the fluoride concentration in the solution while the solution was stirred with a poly (tetrafluroethylane)-coated magnetic bar. Standard curves between approximately 0.04 ppm and 10 ppm were used to calibrate the electrode. An experimental curve of relative millivolts as a function of fluoride concentration was generated by use of various buffered dilutions of the standard solution (Orion Research, Inc). The amount of fluoride measured was converted into micrograms of F^- released per unit specimen area (μg/cm2) according to previous methods (Fukazawa, Matsuya & Yamane, 1990). The cumulative amount of fluoride release was then calculated after each storage period (Mitra, 1991; Verbeeck & others, 1993).

One-way ANOVA was performed to detect significance (a = 0.05) in material properties. Tukey's multiple comparison test was used at a family confidence coefficient of 0.95 to group and rank the measured values.

RESULTS

Figure 1 shows the flexural strength of the four control materials and the whisker composites at whisker/(whisker + fluorosilicate glass) mass fractions of 0.5, 0.7, 0.8, 0.9, and 1.0. For each whisker composite, the

Table 2: *Cumulative Fluoide Release per Specimen Area (μg/cm²) (mean* ± SD; n = 4)*

Whisker/[whisker + fluorosilicate glass] (mass fraction)	1 day	7 days	14 days	30 days	60 days	90 days
0.5	1.6±0.6	3.5±0.1	5.6±0.4	12.0±2.6	25.4±1.0	33.0±1.9
0.7	0.4±0.1	1.1±0.1	1.6±0.2	2.2±0.4	5.6±0.9	11.1±0.8
0.8	2.3±0.7	3.5±1.0	3.7±1.0	3.9±1.1	5.0±1.0	7.1±0.4
0.9	0.7±0.1	0.9±0.1	1.0±0.1	1.1±0.1	1.3±0.3	1.5±0.1
control: traditional glass ionomer	3.7±1.8	16±1.7	23.1±2.3	30.1±1.3	42.3±1.9	53.0±3.4
control: resin-modified glass ionomer	5.7±1.2	16.4±1.2	22.9±1.6	32.5±2.5	47±3.6	65.5±2.8
control: compomer	3.3±1.6	8.0±2.1	11.1±4.2	18.0±5.0	31.3±7.0	39.0±7.5

*Resin-modified glass ionomer had the highest fluoride release followed by glass ionomer and compomer. Among whisker composites, fluoride release was the highest at whisker/[whisker = fluorosilicate glass] of 0.5 (Tukey's multiple comparison test; family confidence coefficient = 0.95). Fluoride release of at whisker/[whisker + fluorosilicate glass] of 1.0 and the composite resin control was not detectable.

mass fraction of resin/(resin + MEP) was equal to that of the whisker/(whisker + fluorosilicate glass)."W/(W + FS)" designates "whisker/(whisker + fluorosilicate glass) mass fraction." In each bar graph, the first bar shows the strength of the specimens without water immersion, and the second bar shows the strength of specimens fractured after 24 hours of immersion in water at 37°C. The two bars in each plot with a horizontal line show strength values that are not significantly different ($p>0.1$; Student's t-test); the two bars in each plot without a horizontal line show values that are significantly different ($p<0.05$; Student's t-test). The flexural strength of all the whisker composites, the compomer, and the composite resin control did not decrease significantly after the one day of immersion in water, while that of the glass ionomer and the resin-modified glass ionomer decreased significantly during the water immersion.

Tables 1A and 1B show flexural properties of specimens without water immersion and after one day of immersion, respectively. Each value is mean with one standard deviation of six measurements. In each column, values with six repeats were statistically compared (Tukey's multiple comparison test; family confidence coefficient = 0.95). The flexural strength of the whisker composite increased with increasing of the mass fraction of whisker/(whisker + fluorosilicate glass), both without water immersion and with one day of immersion. The flexural strength of the whisker composites at whisker/(whisker + fluorosilicate glass) mass fractions of 1.0 and 0.9 was the highest; those at whisker/(whisker + fluorosilicate glass) mass fractions of 0.8 and 0.7 and the control composite resin were statistically similar; and those of the resin-modified glass ionomer and the traditional glass ionomer were the lowest.

Without water immersion, the traditional glass ionomer had the highest elastic modulus, followed by the whisker composites and the posterior composite control, then by the compomer and the resin-modified glass ionomer. After one day of immersion in water, the modulus of the traditional glass ionomer and the whisker composites became generally similar to each other. The work-of-fracture was the highest for the whisker composites at whisker/(whisker + fluorosilicate glass) mass fractions of 1.0 and 0.9, both without water immersion and with one day of immersion. The traditional glass ionomer and the resin-modified glass ionomer had the lowest work-of-fracture values.

Results of the cumulative fluoride-release measurements are listed in Table 2. Each value is the mean with one standard deviation of six measurements. In each column, values with four repeats were statistically compared (Tukey's multiple comparison test; family confidence coefficient = 0.95). The fluoride release of the control composite resin and the whisker composite at a whisker/(whisker + fluorosilicate glass) mass fraction of 1.0 were not detectable. The amount of fluoride release of the glass ionomer and the resin-modified glass ionomer were generally not significantly different. Fluoride release of the whisker composite at a whisker/(whisker + fluorosilicate glass) mass fraction of 0.5 and the compomer control were not significantly different. Figure 2 plots the amount of fluoride release versus storage time for these materials. "RMGI" designates the resin-modified glass ionomer, "GI" designates the glass ionomer, and "W/(W + FS)" designates "whisker/(whisker + fluorosilicate glass) mass fraction," while the mass fraction of resin/(resin + MEP) = whisker/(whisker + fluorosilicate glass).

DISCUSSION

Results of the present study suggest that ceramic whisker reinforcement can substantially improve the mechanical properties of direct restorative materials formulated to release fluoride. Fusing silica glass particles onto the surfaces of individual whiskers not only facilitates whisker silanization, but may have also improved the whisker retention in the matrix by providing rougher whisker surfaces. In addition, two other

factors may have contributed to the efficacy of composite reinforcement. The first factor is the high strength and toughness of the ceramic whiskers. The single-crystalline whiskers have few structural defects and hence tensile strength values as high as 50 GPa (Iwanaga & Kawai, 1998). In comparison, the strength of glass fibers is approximately 3 GPa and that of polished bulk glass is about 0.1 GPa (Lawn, 1993). The fracture toughness of crystalline ceramics (silicon nitride, alumina, zirconia, etc) ranges from about 2 to 6 MPa•m1/2, while that of glass is only about 0.7 MPa•m1/2 (Lawn, 1993). It was observed that while a microcrack can easily cut through a glass filler, the ceramic whiskers are more resistant to fracture (Xu & others, 1999a). The second factor that may have contributed to the reinforcement efficacy is the highly elongated shapes of the whiskers (eg, a diameter of 0.4 μm and a length of 5 μm). Elongated fillers have been observed to be more effective than particulate fillers in bridging a microcrack resisting it from propagating (Lawn, 1993; Xu & others, 1998) and in resisting dislodgement from the matrix. The size of the whiskers are orders of magnitude smaller than most glass fibers and ceramic fibers (eg, a diameter of 10 μm to 100 μm and a length of 100 μm to 1 mm) (Lawn, 1993; Xu & others, 1994). These small whiskers allow a relatively uniform distribution in the matrix with an acceptable composite polishability (Xu & others, 1999b).

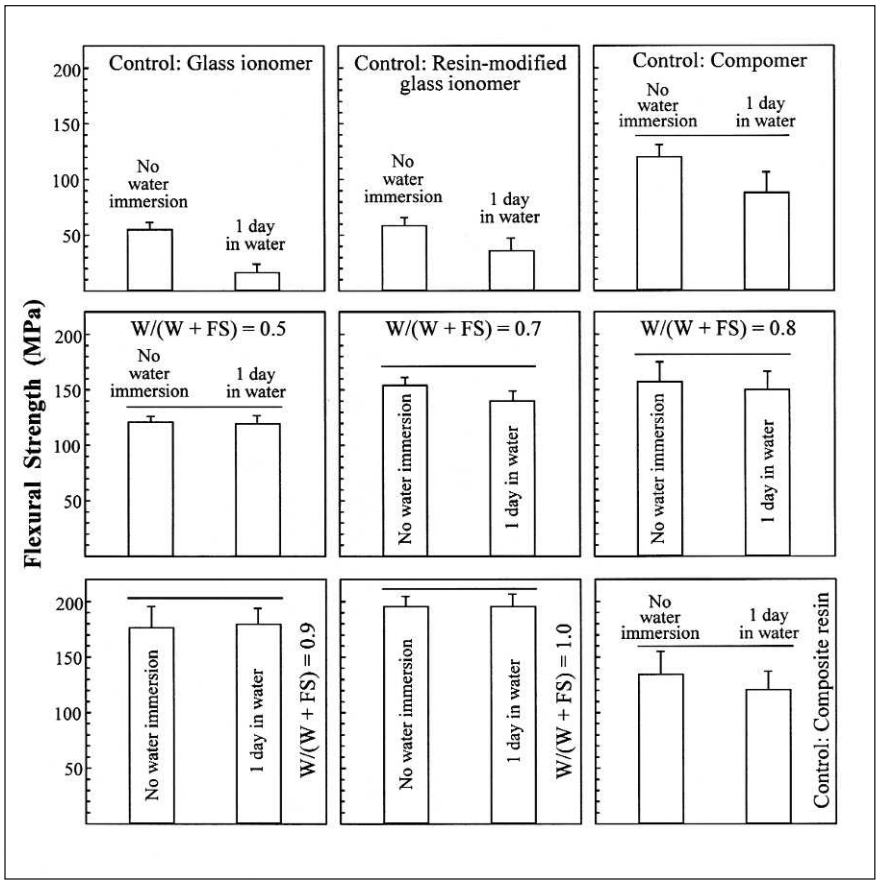

Figure 1. *Flexural strength (mean ± sd; n = 6) of the four control materials and the whisker composites at whisker/(whisker = flurosilicate glass) mass fractions of 0.5, 0.7, 0.8, 0.9, and 1.0. "W/(W = FS)" designates whisker/(whisker = flurosilicate glass) mass fraction. In each plot, the first bar is strength of specimens without water immersion, and the second bar is strength of specimens after a one-day immersion in water. Horizontal lines indicate strength values that are statistically similar (p> 0.1; Student t). Only the glass ionomer and the resin-modified glass ionomer had a significant strength loss after a one-day water immersion.*

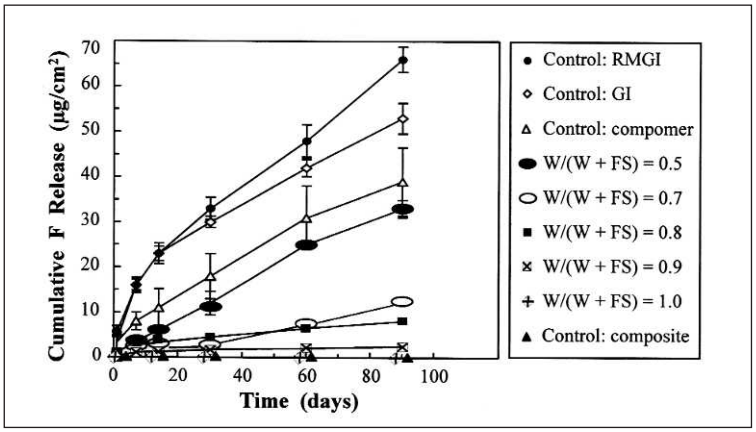

Figure 2. *Fluoride release (mean ± sd; n = 4) versus storage time in distilled water of the four control materials and the whisker composites at whisker/(whisker = fluorosilicate glass) mass fractions of 0.5, 0.7, 0.8, 0.9, and 1.0. "RGMI" designates resin-modified glass ionomer, "GI" designates glass ionomer, and "W/(W = FS)" designates whisker/(whisker + flurosilicate glass) mass fraction.*

Combining ceramic single-crystalline whiskers with fluorosilicate glass in resins modified by the presence of a carboxylic acid monomer in the present study have yielded restorative composites with superior strength that are still able to release fluoride. Compared with the hybrid composite resin control, which did not have fluoride release, the whisker-fluorosilicate composites not only released fluoride, but also had flexural strength values 60 MPa and 30 MPa higher, at whisker/(whisker + fluorosilicate glass) mass fractions of 0.9 and 0.8, respectively. The flexural strength values of the composite resin control, the glass ionomer, and the resin-modified glass ionomer measured in the present study were in the range of those reported in previous studies on

similar materials (Prosser & others, 1986; Mathis & Ferracane, 1989; Mitra & Kedrowski, 1994; Ferracane & Mitchem, 1994). The flexural strength values of the whisker composites were a few times higher, and the work-of-fracture values were orders of magnitude greater than those of the traditional glass ionomer and the resin-modified glass ionomer. The amount of fluoride release by the whisker-fluorosilicate composites was less than that of the traditional glass ionomer and the resin-modified glass ionomer. The minimum amount of fluoride release required to effectively inhibit secondary caries is yet to be determined clinically. A previous study (Swift, 1989b) showed that the fluoride concentration of a fluoride-releasing composite (Heliomolar) was only about 4% of that of a traditional glass ionomer (Ketac-Fil). However, significantly less demineralization was observed in teeth restored with Heliomolar (Arends, Ruben & Dijkman, 1990). The whisker composites of the present study had a cumulative fluoride release after 90 days of storage as high as 62% of that of a traditional glass ionomer, depending on the whisker/(whisker + fluorosilicate glass) mass fraction. Further studies should investigate the fusion of fluorosilicate glass particles, instead of silica, onto the whiskers, thereby possibly increasing the amount of fluoride release of the whisker composites while maintaining the superior mechanical properties.

The present study investigated the mechanical properties of composite specimens without water immersion and with one day of immersion in water at 37°C. Further studies should evaluate the long-term water-aging behavior of these composites, including water absorption, solubility, and durability of the mechanical properties. Ongoing studies on selected whisker composites indicate that the composite strength decreased after prolonged water aging, similar to that of current composite resins. Additional research is required to systematically understand the reinforcement mechanisms and to improve the water-aging resistance of the whisker-reinforced resin-carboxylic composites. Further studies should also aim at approximating the refractive index values of the phases to improve the translucency and esthetics of the composites. In addition, the wear behavior of the composites against enamel has yet to be investigated.

CONCLUSIONS

Ceramic whiskers can significantly reinforce composites containing fluorosilicate glass in a matrix formed from a resin that was modified with a carboxylic acid monomer. The composite mechanical properties and the amount of fluoride release can be adjusted for specific applications by tailoring the whisker:fluorosilicate glass ratio and the resin:carboxylic acid monomer ratio. The sizes of the whiskers are orders of magnitude smaller than available glass fibers, allowing for a relatively uniform distribution in the matrix. Depending on the ratio of whisker:fluorosilicate glass and resin:carboxylic acid monomer, the whisker composite releases fluoride up to 60% of that of a traditional glass ionomer. The flexural strength and work-of-fracture (or toughness) values of the whisker-fluorosilicate composites are significantly higher than those of a traditional glass ionomer, a resin-modified glass ionomer, and a compomer.

Acknowledgments

The authors gratefully thank H Y Liao for assistance and Drs S H Dickens and S Takagi for discussions on fluoride-release measurements. This study was supported by NIDR grants P 50 DE09322 and R 29 DE12476, NIST, and the ADAHF.

Disclaimer

Certain commercial materials and equipment are identified in this paper to specify experimental procedures. In no instance does such identification imply recommendation by NIST or the ADA Health Foundation or that the material identified is necessarily the best available for the purpose.

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Polymerization Shrinkage of Visible-Light-Cured Composites

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KS Siow • LM Gan

Clinical Relevance

Conventional and polyacid-modified composite resin shrinkage continues after removal of the curing light and is greatest at one hour. Composite restorations, if polished immediately after light curing, should be rechecked after one month to determine if repolishing is required.

SUMMARY

This study investigated the long-term dimensional changes of a conventional and a polyacid-modified composite resin and the effects of hydration on polymerization shrinkage. A strain-monitoring device was used to measure the linear polymerization shrinkage of the composites in the free state when stored in water at 37°C or air at 26°C over a one-month period. Results showed that the polymerization reaction of both conventional and polyacid-modified composite resins was accompanied by a dimensional shrinkage change. The rate of shrinkage for both composites was greatest during the polymerization reaction and continued after removal of the curing light. When stored in water, the greatest shrinkage was noted at one hour for both materials. This was followed by a slow uptake of water and expansion from one day to one month. The polyacid-modified composite had significantly less polymerization shrinkage than the conventional composite after one month of storage in water.

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INTRODUCTION

Both the general reduction of dental caries and patient interest in dental aesthetics have resulted in the development of new tooth-colored restoratives and techniques. Composite materials and adhesive techniques have become the foundation of modern dentistry, and composites are one of the most heavily researched dental materials today. Mechanical performance, wear resistance, and aesthetic potentials of composite resins have been significantly improved over the last few years, and composites are now used in cases ranging from the restoration of initial decay and cosmetic corrections to veneering in complex prosthodontic rehabilitation.

Polymerization shrinkage of composite resins, however, remains a challenge and still imposes limitations in the application of direct techniques (Dietschi & Dietschi, 1996). The total shrinkage can be divided into two components: the pre-gel and post-gel phases. During the pre-gel polymerization, the composite is

able to flow, which relieves stress within the structure (Davidson & de Gee, 1984). After gelation, flow ceases and cannot compensate for shrinkage stresses. Post-gel polymerization, therefore, results in clinically significant stresses in composite-tooth bond and surrounding tooth structure (Feilzer, de Gee & Davidson, 1987). Stresses arising from post-gel polymerization shrinkage may produce defects in the composite-tooth bond, leading to bond failure and microleakage with associated postoperative sensitivity (Eick & Welch, 1986) and recurrent caries. Such shrinkage stresses could also cause deformation of the surrounding tooth structure if the composite-tooth bond is good (Sheth, Fuller & Jensen, 1988). The resulting coronal deformation may result in postoperative sensitivity and microcracks in the cervical enamel (Bowen, Nemoto & Rapson, 1983), which predisposes the tooth to fracture.

Although polymerization shrinkage of composite resins has been extensively investigated, few studies have been conducted on polyacid-modified composite resins and the long-term dimensional changes in resin composites. Sakaguchi and others (1991) reported a method of measuring post-gel polymerization shrinkage in composite resins using electrical resistance strain gauges. The model described in this paper utilizes this technology to investigate the long-term dimensional changes of a conventional and a polyacid-modified composite resin. The effect of hydration on polymerization shrinkage was also studied.

Table 1: *Mean shrinkage and standard deviation for Spectrum TPH*

Time (t)	In water at 37°C Linear shrinkage (%)	In air at 26°C Linear shrinkage (%)
t = 0	0	0
t = 20 sec.	-0.18 (±0.02)	-2.21 (±0.02)
t = 10 min.	-0.36 (±0.02)	-0.39 (±0.01)
t = 1 hr.	-0.43 (±0.02)	-0.44 (±0.02)
t = 1 day	-0.41 (±0.03)	-0.48 (±0.02)
t = 3 days	-0.39 (±0.05)	-0.52 (±0.00)
t = 1 week	-0.33 (±0.06)	-0.53 (±0.00)
t = 2 weeks	-0.27 (±0.09)	-0.53 (±0.00)
t = 1 month	-0.20 (±0.10)	-0.52 (±0.00)

Curing light was turned on at t = 0 secs. and off at t = 20 secs.

Table 2: *Mean shrinkage and standard deviation for Dyract AP*

Time (t)	In water at 37°C Linear shrinkage (%)	In air at 26°C Linear shrinkage (%)
t = 0	0	0
t = 40 sec.	-0.22 (±0.02)	-2.24 (±0.03)
t = 10 min.	-0.37 (±0.02)	-0.41 (±0.02)
t = 1 hr.	-0.48 (±0.02)	-0.45 (±0.02)
t = 1 day	-0.45 (±0.03)	-0.51 (±0.01)
t = 3 days	-0.37 (±0.07)	-0.54 (±0.02)
t = 1 week	-0.27 (±0.06)	-0.53 (±0.02)
t = 2 weeks	-0.16 (±0.03)	-0.53 (±0.02)
t = 1 month	+0.20 (±0.04)	-0.47 (±0.02)

Curing light was turned on at t = 0 secs. and off at t = 40 secs.

METHODS AND MATERIALS

A conventional hybrid composite resin (Spectrum TPH; Dentsply, Milford, DE 19963) and a polyacid-modified composite resin (Dyract AP; Dentsply) of the same shade (A2) were selected for this study. The test configuration used a stiff silicone frame (inner length 10.0 mm, width 5.0 mm, and height 2.0 mm), which circumscribed the composite sample (Figure 1). A glass slide served as the base of the set-up. A foil electrical resistance strain gauge (Foil Strain Gauge; RS Components Ltd, Singapore) was attached onto the flat glass surface. The gauge was 2 mm in length and had an electrical resistance of 120 W and gauge factor of 2.00. The composite resins were placed in the cavity of the silicone frame with the strain gauge in place. Care was taken to ensure complete filling of the frame, and the excess composite material was extruded using pressure applied through a second glass slide and removed. The surface tack of the composite was adequate to ensure adhesion between the strain gauge and the composite

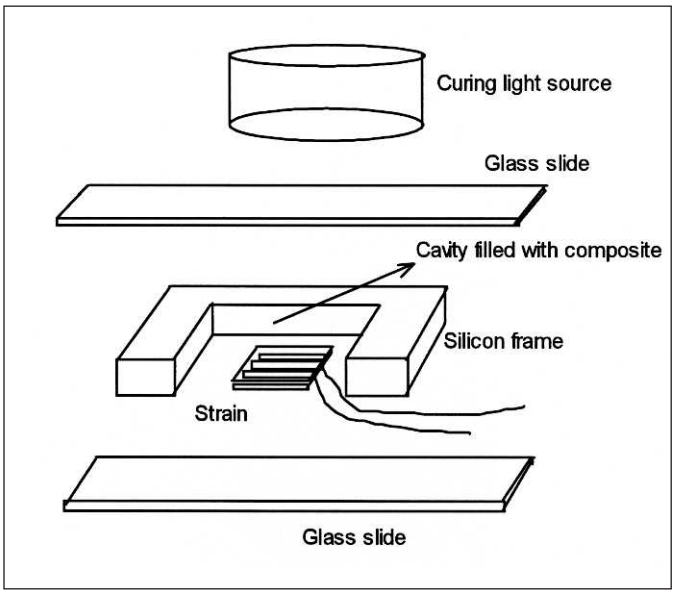

Figure 1. *Schematic drawing of the experimental set-up.*

materials. The leads from the strain gauge were connected to a strain monitoring device (Strain Gauge Recorder; Cole Parmer Instruments, Vernon Hills, IL 60061) initially balanced at zero.

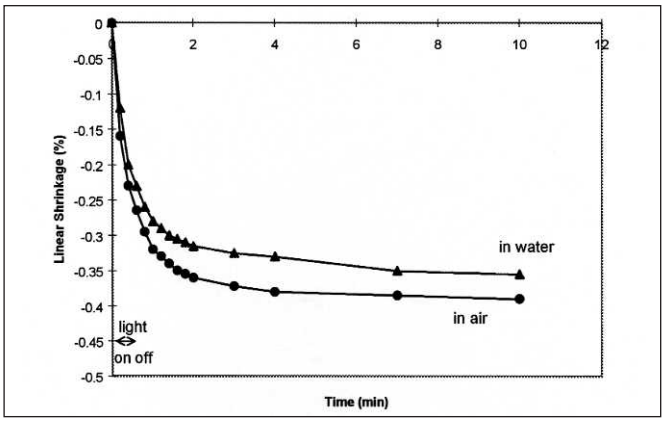

Figure 2. *Polymerization shrinkage of Spectrum TPH during the initial 10 mins.*

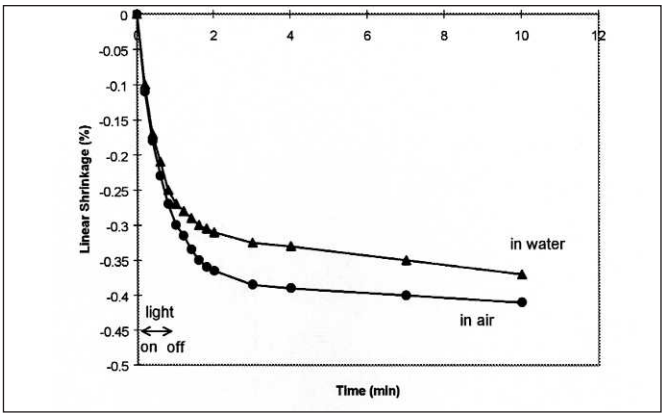

Figure 3. *Polymerization shrinkage of Dytract AP during the initial 10 mins.*

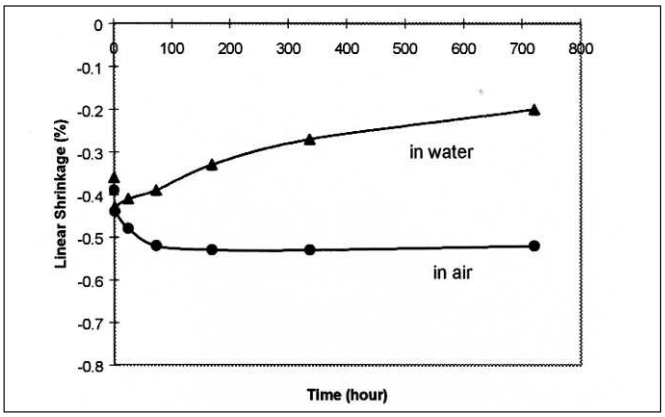

Figure 4. *Shrinkage during the aging process for Spectrum TPH.*

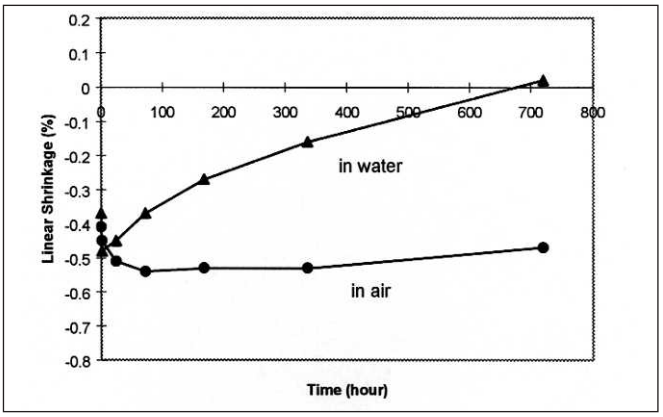

Figure 5. *Shrinkage during the aging process for Dyract AP.*

The composite specimens were light polymerized (HILUX 250; Benlioglu Dental Inc, Ankara, Turkey), and the dimensional change during the light-polymerization period was monitored. The cure time was 20 seconds for Spectrum TPH and 40 seconds for Dyract AP, as recommended by the manufacturer. The silicone frame and glass plates were removed immediately after polymerization. The composite specimens were kept in a free state and stored in either distilled water at 37°C or left in air at room temperature (26 ± 1°C). Polymerization shrinkage measurements were taken continuously every 0.2 minutes up to 10 minutes. The specimens were then aged up to a period of one month in water at 37°C or air at room temperature, and the change of resistance of the strain gauge was recorded during the aging process at the following time intervals: one hour, one day (24 hours), three days (72 hours), one week (168 hours), two weeks (336 hours), and one month (720 hours). The percentage linear shrinkage was derived from the following equation: Percentage linear shrinkage (DL/L x 100) = (DR/R)/K x 100; where DL = change in length; L = original length; DR = change in resistance; R = original resistance; K = gauge factor (ie, 2).

Multiple trials (n=3) were performed for each of the composite-medium combination. The data were subjected to one-way ANOVA and Scheffé's multiple range tests at a 0.05 significance level to determine significant changes in shrinkage with time. An independent t-test was used to investigate the effects of hydration on polymerization shrinkage and to compare shrinkage between the two composites.

RESULTS

The mean percentages of linear shrinkage of Spectrum and Dyract at the various time intervals are reflected in Tables 1 and 2, respectively. The shrinkage during the initial 10 minutes is shown in Figures 2 and 3. Figures 4 and 5 show the dimensional changes associated with long-term aging in water and air.

The rate of shrinkage for both Spectrum and Dyract was greatest during the light-polymerization reaction and continued after the curing light was removed. For both composites stored in water, the rate of shrinkage continued to be high postpolymerization and stabilized at about one hour. This was followed by an expansion of about 0.02 to 0.03% at one day. Both materials contin-

ued to expand up to one month. At one month, Spectrum had a residual shrinkage of -0.20%, while Dyract had an expansion of 0.02%. For Spectrum aged in water, shrinkage at one hour and one day was significantly greater than that at immediate postpolymerization (20 seconds) and one month. For Dyract aged in water, the dimensional change at one month was significantly different from that at all other time periods. In addition, the shrinkage at one hour and one day was significantly greater than that at one week. The linear shrinkage of Spectrum was significantly greater than that of Dyract only after one month of storage in water.

When aged in air, both Spectrum and Dyract showed a rapid increase in shrinkage postpolymerization. The dimensional change of both materials stabilized and was maintained after three days. For Spectrum in air, shrinkage postpolymerization (20 seconds) was significantly less than those at all time periods. Shrinkage at 10 minutes was significantly smaller than that from one day onwards, and shrinkage at one day was significantly less than that at three days, one week, two weeks, and one month. For Dyract in air, shrinkage postpolymerization (40 seconds) was also significantly less than that at all other time periods. Percentage of linear shrinkage at 10 minutes was significantly smaller than that at one day, three days, one week, and two weeks.

DISCUSSION

Polyacid-modified composite resins or compomers are a new class of restorative materials that combine fluoride release and good biocompatibility with the easy handling and aesthetic property of composite resins. They contain the reactive fluorosilicate glass of conventional glass-ionomer cements, but their initial setting is due to a light-activated radical polymerization. After the initial light-activated polymerization, the traditional glass-ionomer reaction slowly emerges through the uptake of water and the establishment of an acid-base reaction, resulting in a partially ionic structure within the polymeric matrix (Dentsply, 1994). Fluoride release (Yap, Khor & Foo, 1998) and possible chemical adhesion in a similar manner to that of glass ionomers is thus achieved.

As seen from the results of this study, the polymerization reaction was accompanied by a dimensional change that resulted in shrinkage for both types of composites. Shrinkage was caused by the monomers becoming covalently bonded by the polymerization reaction, thus exchanging van der Waals' distances for covalent bond distances. The degree of shrinkage is dictated by the number of covalent bonds formed, ie, the extent of the polymerization reaction, as well as the sizes of the monomers (Ferracane, 1995). Other possible influencing factors were the amount of filler, filler type, size, and coating network (Feilzer, de Gee & Davidson, 1988). The resin matrix of Spectrum consists of a BIS-GMA adduct, ethoxylated bisphenol-A-dimethacrylate (BIS-EMA), and triethylene glycol dimethacrylate. The filler system used in Spectrum is a combination of barium aluminoborosilicate glass with a mean particle size below 1 mm (maximum 5 mm) and colloidal silica (particle size 0.04 mm). The total percentage by weight of inorganic fillers is 77%. The resin matrix of Dyract AP, however, consists of urethane dimethacrylate and TCB resin, which is a reaction product of tetracarboxylic acid and HEMA (hydroxyethylmethacrylate). Reactive strontium fluorosilicate glass (particle size 0.8 mm) accounts for up to 72% weight of the composition.

The rate of shrinkage for Spectrum was highest during light polymerization (Figure 2). When the curing light was turned off at 20 seconds, the rate of shrinkage decreased but was still substantially high (-0.15%/min) up to one minute, when the rate dropped to –0.05%/minute. The rate of shrinkage for Dyract was also highest during light polymerization (Figure 3). When the curing light was turned off at 40 seconds, the rate of shrinkage decreased to -0.1 to -0.15%/minute for Dyract stored in water and air, respectively. At one minute the shrinkage rate had dropped to -0.05%/minute for specimens stored in water and -0.075%/minute for specimens stored in air. At two minutes, both Spectrum and Dyract had similar linear shrinkage for both specimens stored in water and air.

The shrinkage noted after removal of the light source may be partially attributed to thermal contraction due to loss of radiant heat. This phenomenon was observed for Spectrum and Dyract specimens stored in both water and air. The high shrinkage rate during the first one minute may be clinically significant, as the integrity of the tooth-composite interface is rapidly challenged during the early phases of polymerization, when the bond between enamel or dentin and the composite is still maturing. The rate of shrinkage continued to decrease with time and at 10 minutes postpolymerization, the shrinkage of Spectrum and Dyract when soaked in water was –0.36% and –0.37%, respectively. The shrinkage in air was –0.39% for Spectrum and –0.41% for Dyract. Polishing procedures should therefore be delayed, as composites may not be dimensionally stable immediately following light curing.

When stored in water, the shrinkage of Spectrum was maximum at one hour. This was followed by an expansion from day one to one month, when a residual shrinkage of only -.20% was observed. In contrast, Spectrum specimens stored in air showed a progressive shrinkage, which stabilized at day three. At one month, the residual shrinkage of specimens in air remained at –0.52%. The expansion noted with storage in water could, therefore, be attributed to the water sorption by the composite.

Although the shrinkage and build-up of internal stresses are very rapid and take place within minutes in Spectrum, water sorption occurs at a much slower rate. This finding is consistent with that of Ferracane and Condon (1990), who reported that water uptake by composite resin requires hours or days to reach saturation. The delayed nature of water sorption thus minimizes its effects on stress reduction. In addition, water sorption may cause erosion of the filler/matrix interface and a softening of the polymer network, which may result in decreased mechanical properties and wear resistance (Söderholm & Roberts, 1990). Water sorption is reduced by the use of more hydrophobic monomers like the exthoxylated version of BIS-GMA (BIS-EMA) used in Spectrum, which does not contain unreacted hydroxyl groups on the main polymer chain.

For Dyract stored in water, shrinkage was also maximum at one hour. This was followed by an expansion up to one month, when a residual expansion of 0.02% was noted. As progressive shrinkage was noted with Dyract in air up to day three, the expansion observed with storage in water can only be attributed to the effects of hydration as for Spectrum. The uptake of water by Dyract occurred at a much faster rate than in Spectrum and started after one week storage in water. A significant difference in dimensional change existed between Spectrum and Dyract only after one month of storage in water. The greater water uptake by Dyract may be attributed to the presence of HEMA in TCB resin. Copolymers of HEMA can take up large amounts of water, possibly up to 80% by mass (Pedley, Skelly & Tighe, 1980). This uptake of water is necessary for the activation of the acid-base reaction within the polymer matrix.

All factors being equal, a resin composite demonstrating lower polymerization shrinkage would clinically be preferred. Although the initial shrinkage of both composites stored in water is similar, the greater water sorption of Dyract resulted in an expansion at one month, which theoretically leads to a better marginal seal. Other factors, such as wear rate, fracture resistance, and fluoride release, however, need to be considered. The technique of composite placement also needs to be carefully considered to minimize the effects of polymerization shrinkage. The bulk placement technique used in this experiment should not be encouraged in a clinical setting, although new composite restoratives have been formulated for bulk placement of up to 5 mm. The net effects on the surrounding tooth structure may be minimized through incremental placement of composite resin (Lutz, Krejci & Oldenburg, 1986) as recommended by most composite manufacturers. Although there is now some controversy about whether incremental placement provides better marginal adaptation (Versluis & others, 1996; Winkler, Katona & Paydar, 1996), it is still necessary to provide adequate light curing when composite thickness exceeds 2 mm (Caughman Rueggeberg & Curtis, 1995).

CONCLUSIONS

Under the conditions of this *in vitro* study:

1. The polymerization reaction of both conventional and polyacid-modified composite resins was accompanied by a dimensional change that results in shrinkage.

2. The rate of shrinkage for both composites was greatest during the polymerization reaction and continued after the curing was completed.

3. When stored in water, the greatest shrinkage was noted at one hour for both materials. A slow water uptake and expansion followed this from one day to one month.

4. The polyacid-modified composite had significantly less polymerization shrinkage than the conventional composite after one month of storage in water.

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Fluoride Uptake Around Cavity Walls; Two-Dimensional Mapping by Electron Probe Microanalysis (EPMA-WDX) Method

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Clinical Relevance

Two-dimensional mapping of fluoride uptake around cavity walls could provide useful information in the development of new fluoride-releasing restorative materials.

SUMMARY

This study reports fluoride uptake around the cavity wall of teeth by two-dimensional mapping. Fluoride concentration was measured using the wavelength dispersive x-ray analysis (WDX) method. The buccal cavity wall of a human tooth was coated five times with 2% sodium fluoride solution at three-day intervals for 12 days, and then immersed in a normal saline solution at 37°C. After one month, the tooth was bisected longitudinally through the center of the cavity surface perpendicular to the axial wall. On the polished surface of the cut tooth, the fluoride concentration was measured. Fluoride distribution maps around the cavity wall were drawn using a bundle of the observed analytical lines. Fluoride uptake from fluoride-releasing materials (conventional glass-ionomer cement, light-cured glass-ionomer cement, light-cured composite resin, light-cured bonding agent) around the cavity wall was investigated using the same method. The maps showed higher fluoride uptake in dentin than in enamel and a strong location dependence of fluoride uptake in a tooth, especially in the dentin. Fluoride uptake from the resin was greater than that from the cement. It was summarized from these results that a two-dimensional map of fluoride uptake can provide valuable information on the cariostatic properties of fluoride-releasing materials.

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INTRODUCTION

Fluoride exhibits cariostatic properties on hydroxyapatite (Brown, Gregory & Chow, 1977) and has been clinically applied in such forms as fluoride varnish (de Bruyn, Buskes & Arend, 1986), fluoride toothpastes (ten Cate & others, 1988), and mouth rinses (Mellberg

& others, 1988). Besides the glass-ionomer cement in which fluoride is necessarily included through the fabrication process, restorative materials containing fluoride additives have been intensively developed recently for the gradual release of fluoride (Kadoma, Kojima & Masuhara, 1983; Dijkman & others, 1993; Park & Kim, 1997). Fluoride uptake by teeth is one of the criteria for evaluation of the cariostatic properties of fluoride. Various papers have reported on the uptake of fluoride into teeth from the above-mentioned materials. In most of these studies, the experiments were carried out using the flat surfaces of teeth as specimens (de Bruyn, Hummel & Arends, 1985; Temin, Csuros & Mellberg, 1989; Tsanidis & Koulourides, 1992). Only a few papers have reported uptake over a partial part of the cavity wall (Tveit & Tøtdal, 1981; Skartveit & others, 1990; Mukai & others, 1993). Uptake of fluoride around the whole cavity preparation has, to our knowledge, yet to be examined.

Two kinds of methods have been adopted for fluoride measurement in a tooth. One is the chemical method (destructive testing) and the other is physical (nondestructive testing). The former method, after some chemical pretreatment of the specimen, usually measures fluoride as ions in solution using such analytical equipment as fluoride ion-specific electrodes (McCann, 1968; Larsen, Kold & von der Fehr, 1972; Weatherell & others, 1985; Ikemi & Koulourides, 1988) or gas chromatography (Munksgaard & Bruun, 1973; Bruun & Givskov, 1993). Although widely adopted for fluoride measurement, this method has the disadvantage that the original shape of the specimen is destroyed during measurement. Nondestructive testing is possible by using electron probe microanalysis (EPMA) (Besic & others, 1969; Hals & Selvig, 1977; Eliades, Viazis & Eliades, 1992; Yu & others, 1993) and/or proton-induced gamma-ray emission analysis (PIGE) (Nelson & others, 1992; Pearce, Coote & Larsen, 1995; Shu & others, 1998). Both EPMA and PIGE preserve the original shape of the specimen even after measurement, and can identify the location measurement in a specimen. Therefore, the nondestructive testing method may be a useful tool for investigating spacial distribution of fluoride in teeth. Employing this nondestructive analysis for studying fluoride uptake, quantitative uptake of fluoride around a cavity wall in a tooth was reported. The results were twofold. First, the nondestructive method was demonstrated by mapping two-dimensional fluoride distribution around the cavity wall using NaF-solution as a fluoride-releasing material. NaF-solution was chosen for its smooth penetration of fluoride ion into the tooth. Secondly, the mapping of the fluoride and the characterization of clinically used fluoride-releasing materials were estimated using this method.

METHODS AND MATERIALS (MAPPING OF THE NaF TOOTH)

Specimen Preparation

Human premolar teeth were used as specimens. These teeth (18-29 year-old patients) were extracted for orthodontic reasons within two months prior to the study, and had no cracks, white spots, or enamel defects. The cavity positions (denoted "a" and "b") are shown in Figure 1. The first one ("a") was at a position 2.5 mm up from the cementoenamel junction along the median line and 1.5 mm from the median line to the mesial side. The second ("b") was at the same height but on the opposite (distal) side of the median line. The direction of the cavity was toward the pulp. The two cavities had the same size and shape: that of an elliptic pillar (2.0 mm x 1.5 mm x 1.5 mm). The depth of 1.5 mm was sufficient to reach the axial wall of the dentin. The cavities were made with a water-cooled, high-speed handpiece and diamond points (FG202 and HI-C; Shofu Inc, Kyoto, Japan). Sodium fluoride solution (2% Sodium Fluoride [Neo]; Neo Pharmacy Industry, Tokyo, Japan) was applied to cavity ("a") on the mesial side, serving as fluoride-releasing material for four minutes. (Hereafter the material name is referred to as "NaF" and the tooth it was applied to as the "NaF-tooth"). The distal cavity ("b") was used as a control. The tooth was coated with nail polish except for the NaF-applied cavity wall, immersed in a plastic capsule filled with 10 ml of normal saline solution (saline), and kept at 37°C. At intervals of three days, NaF was again applied to the cavity wall and, at the same time, the saline solution was renewed. Following four applications or 12 days, the solvent only was renewed at intervals of one week.

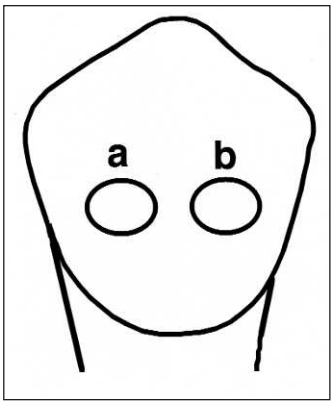

Figure 1. *Cavity preparation. Size: 2.0mm x 1.5mm x 1.5mm. "a" was at height 2.5mm from the enamel-cement junction along the median line and 1.5mm from the median line to the mesial side and applied with NaF. "b" was the same height as "a," but on the opposite side to the median line, and used as a control.*

Fluoride Measurement

After one month of immersion, three NaF-teeth were removed from the saline, embedded in epoxy resin, and bisected longitudinally through the center of the cavity perpendicular to the axial wall with a precision saw (Isomet; Buehler Ltd, Lake Bluff, IL 60044). The surface of the cut tooth was polished with impervious paper from #400 to #4000, successively, followed by 0.05 μm alumina-paste polishing. A thin layer of carbon was

then vapored onto the polished surface for WDX measurement. The effect of damage by cutting and polishing on WDX measurement of fluoride uptake was checked preliminarily, but no effect was observed outside of experimental error.

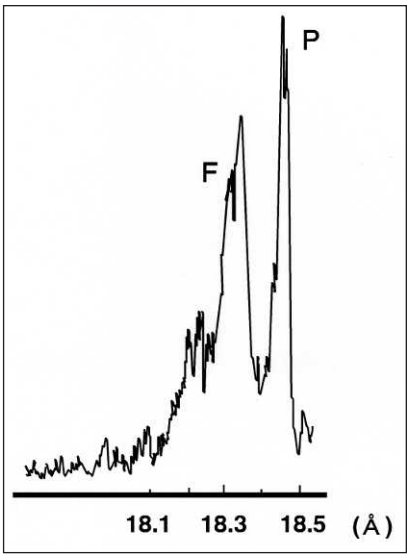

Figure 2. *Separation of Kα line of fluoride and tertiary harmonic of Kα line of phosphate. F: fluoride. P: phosphate.*

On the polished surface, the fluoride concentration around the whole cavity wall was measured using WDX (EPMA-X-650 and Hitachi S2700+Microspec; Hitachi, Ibaragi, Japan). The WDX scans were taken from points on the cavity wall towards the inner part of the specimen (perpendicular to the cavity wall) at about every 300 μm from the line angle. The cutoff point of measurement, beyond which only natural fluoride existed, was determined by observed x-ray intensity. The x-ray count (cps) decreased steadily from the surface towards the inner part, and beyond the cut-off point the observed intensity kept a nearly constant figure, which was considered the base line. The measurement conditions were as follows: accelerating voltage 15kV, probe current 0.05μA, region of x-ray production 1 μm ø x 5 μm, travel speed of specimen 10 μm/minute, time constant 0.2 seconds, analyzing crystal RAP. In terms of line analysis, the Kα line of fluoride (18.32Å) was closely adjacent to the tertiary harmonic of the Kα line of phosphate (18.45Å), and clear peak separation of the two lines was required for the analysis. The condition was fulfilled in the experiment, and one of the observed patterns is shown in Figure 2. A partially fluoridated hydroxyapatite, from which a calibration curve of fluoride concentration versus WDX intensity was constructed, was used in pressured pellet form. The standard samples were made synthetically, in which fluoride exchanged for hydroxyl at 0%, 25%, 50%, 75%, and 100% rates, respectively (Okazaki & others, 1981). The shape of the cavity was also observed by SEM. Contour lines of fluoride concentration were made by connecting the same fluoride concentration point on each analytical line at intervals of 1000 ppm, except for the first and the second lines. The first, which was the outermost contour line from the cavity wall, represented the base line. The second showed a higher concentration–500 ppm–than that of the first.

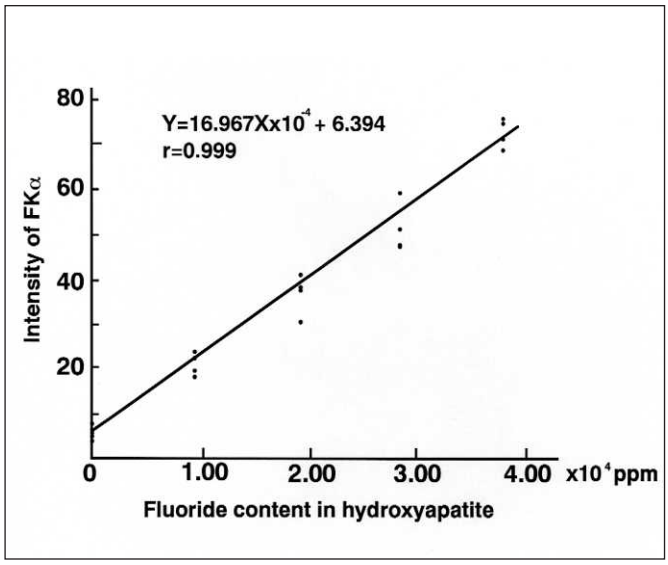

Figure 3. *Linear regression showing the relationship between fluoride concentration and intensity of fluoride Kα line. The fluoridated hydroxyapatites in which fluorine exchanged for hydroxyl at 0, 25, 50, 75, 100% rates were scanned as standard samples.*

Mapping Method

The fluoride concentration (ppm) was estimated using the following formula from the observed x-ray intensity of the Kα line: Y=16.967X x 10^{-4}+6.394; r(reliability coefficient)=0.999; X: fluoride concentration(ppm); Y: x-ray intensity(cps). The calibration curve obtained is shown in Figure 3. After the procedure described above, the contours of uptaken fluoride around the cavity of the tooth, in other words, the map of uptaken fluoride, were obtained using a bundle of the observed analytical lines.

The obtained maps of a NaF-tooth are shown in Figures 4a-c. In the mapping, magnification along the measured line (or perpendicular to the cavity wall) was enlarged 10 times, comparing distance along the cavity wall to stress penetration depth. The location of the measured line in the tooth was confirmed by SEM observation of the electron beam trace of WDX measurement.

The differences in the depth of fluoride uptake and fluoride concentration were analyzed statistically using ANOVA and Fisher's PLSD.

RESULTS
(MAPPING OF THE NaF-TOOTH)

The obtained maps showed characteristic features. There was a strong dependence of fluoride uptake upon the location of the cavity wall from where measurement was done toward the inner part of the tooth, especially in the dentin. Generally, fluoride uptake was higher in dentin than in enamel and less location-dependent in enamel than in dentin. In enamel, the average fluoride concentration at the cavity wall (surface fluoride con-

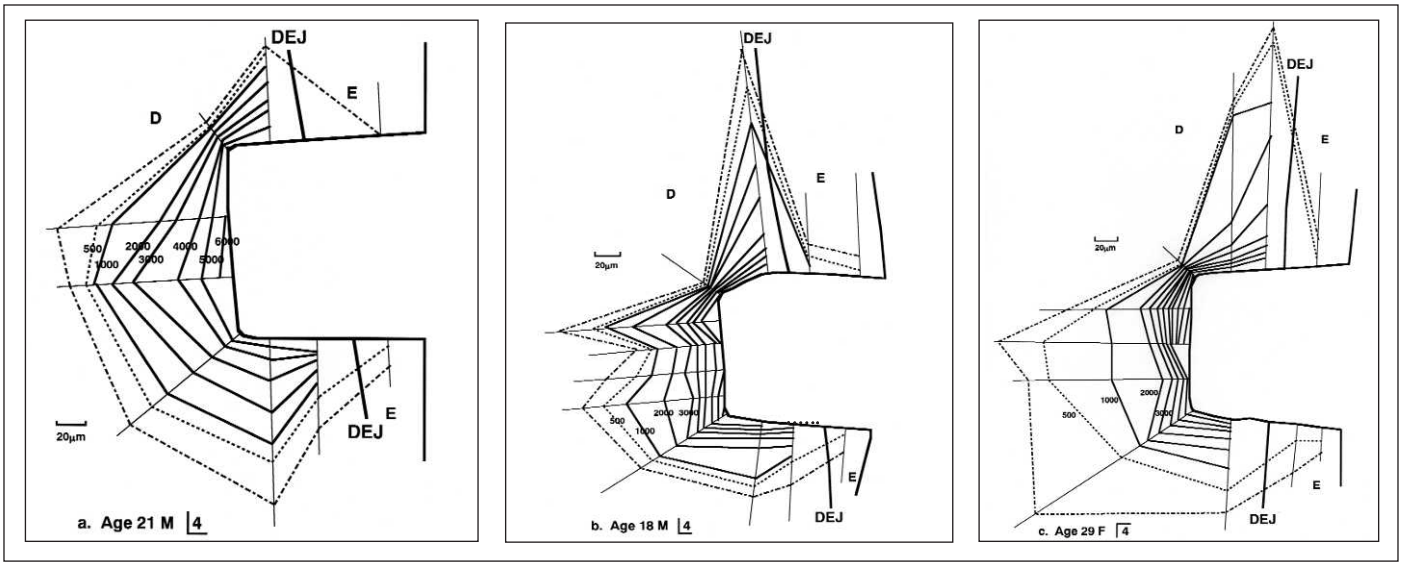

Figure 4 a-c. *Map of fluoride distribution around the entire cavity wall after one month, treated with NaF solution five times over 12 days. Bold line (▬) shows the cavity wall, Fine line (—) shows the places scanned from the surface of the cavity wall toward the inside of the tooth, Medium line (▬) shows each 1000 fluoride ppm unit, Dotted line (·····) shows 500 fluoride ppm, Broken line (- - -) shows baseline, which means a detection limit.*

D;dentin, E;enamel, DEJ; dentin-enamel junction

centration) was around 500 ppm and did not exceed 1000 ppm, whereas in dentin, the surface fluoride concentration was between 5.5 x10^3 ppm and 9.7 x10^3 ppm (side wall) and 5.7 x10^3 ppm and 11 x 10^3 ppm (axial wall), depending on location. The fluoride concentration decreased steeply along the direction perpendicular to the cavity wall, but the rate of decrease was different from location to location on the cavity wall. In other words, the penetration depth of the fluoride was location-dependent in the tooth. The distance from the cavity wall to the base line was defined as the maximal depth of fluoride uptake. The observed figures of surface fluoride concentration and maximal depth of fluoride uptake at the occlusoaxial line angle, side walls, axiogingival line angle, and the axial wall are given in Figures 5A and B. In the enamel, the penetration depth lay between 0 and 15 μm (average 8 μm). In the dentin, the figure was between 48 μm and 215 μm (average 125 μm) at the side walls of cavities and between 66 μm and 219 μm (average 138 μm) at the axial wall. Roughly estimating the maximal depth of fluoride uptake, the exceptional point was along the direction of the occlusoaxial line angle. The surface fluoride concentration at this point was nearly the same as the other locations on the cavity wall (average value 8 x 10^3ppm). Penetration depth was very shallow and the maximal depth of fluoride uptake was near zero beyond around 20 μm. The situation is clearly shown in the maps, with a steep slope of the contour lines.

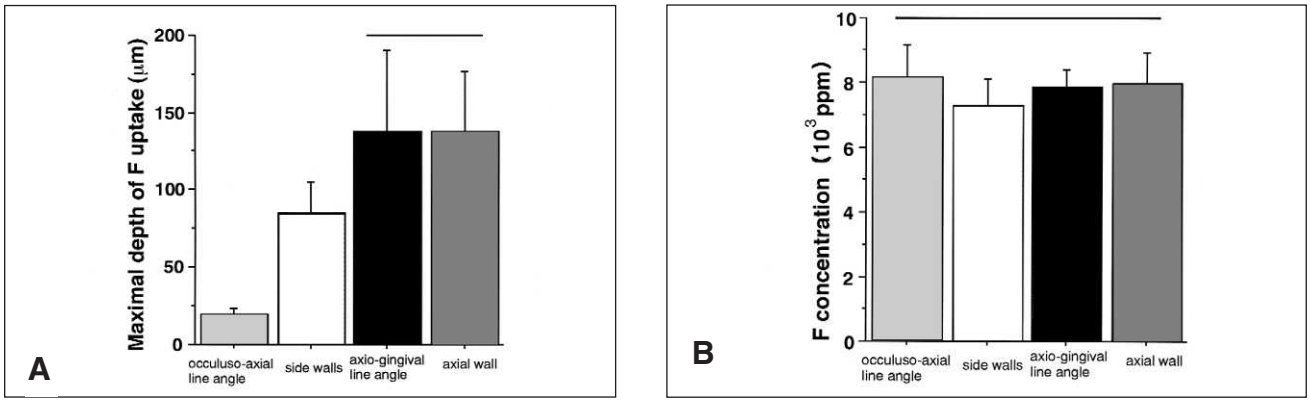

Figure 5. *Fluoride uptake by location on cavity wall in dentin after one month, treated in NaF solution five times over 12 days. (A) The maximum depth of fluoride uptake from the surface of the cavity wall. (B) The fluoride concentration at the surface of the cavity wall. Horizontal line (▬) shows statistically insignificant difference ($p<0.05$).*

METHODS AND MATERIALS
(MAPPING OF TEETH WITH FLUORIDE-RELEASING MATERIALS)

Specimen Preparation

Class V cavities in human teeth (13-29 years old) were prepared as described above. The cavity ("a" in Figure 1) was filled with one of the following fluoride-releasing materials: (a) conventional glass-ionomer cement (Fuji Ionomer Type II; GC Corp, Tokyo, Japan, hereafter abbreviated as TII, and the tooth filled with the material as the TII-tooth); (b) light-cured glass-ionomer cement (Fuji Ionomer Type II LC; GC Corp, abbreviated as LC and LC-tooth); (c) light-cured composite resin (Teethmate F; Kuraray Co, Ltd, Osaka, Japan, abbreviated to TF and TF-tooth); (d) light-cured bonding agent (Kurasper F·F-bond; Kuraray, abbreviated to FB and FB-tooth); as well as nonfluoride-releasing light-cured composite resin (Silux Plus; 3M Dental Products, St Paul, MN 55144). TII powder was mixed with the liquid (powder:liquid=2.7:1), pressurized into the cavity, and the surface area was protected with varnish (GC Fuji varnish; GC Corp). The mixed LC (powder:liquid=3:1) was placed into the cavity preparation and then cured for 40 seconds using a visible light-curing unit (Quick Light; J Morita Corp, Osaka, Japan) through a Mylar matrix and varnished. In the case of TF, the cavity was etched with acid (K-etchant; Kuraray) for 40 seconds, washed, and dried for 15 seconds. TF was inserted into the cavity and cured for 40 seconds. For FB, the cavity was etched with K-etchant for 40 seconds. After washing and drying for 15 seconds, FB was inserted and cured for 10 seconds, then Silux Plus was placed and cured for 40 seconds. Three teeth were used for each of the fluoride-releasing materials. Each tooth was coated with nail polish except for the surface of the filling material. After 30 minutes the teeth were immersed in a plastic capsule filled with 10 ml saline and kept at 37°C. The solvent was renewed at intervals of one week. After immersion for one month, pretreatment of cutting, polishing, and coating of teeth were performed as before.

Mapping Method

The fluoride measurement and mapping procedure were performed as described above.

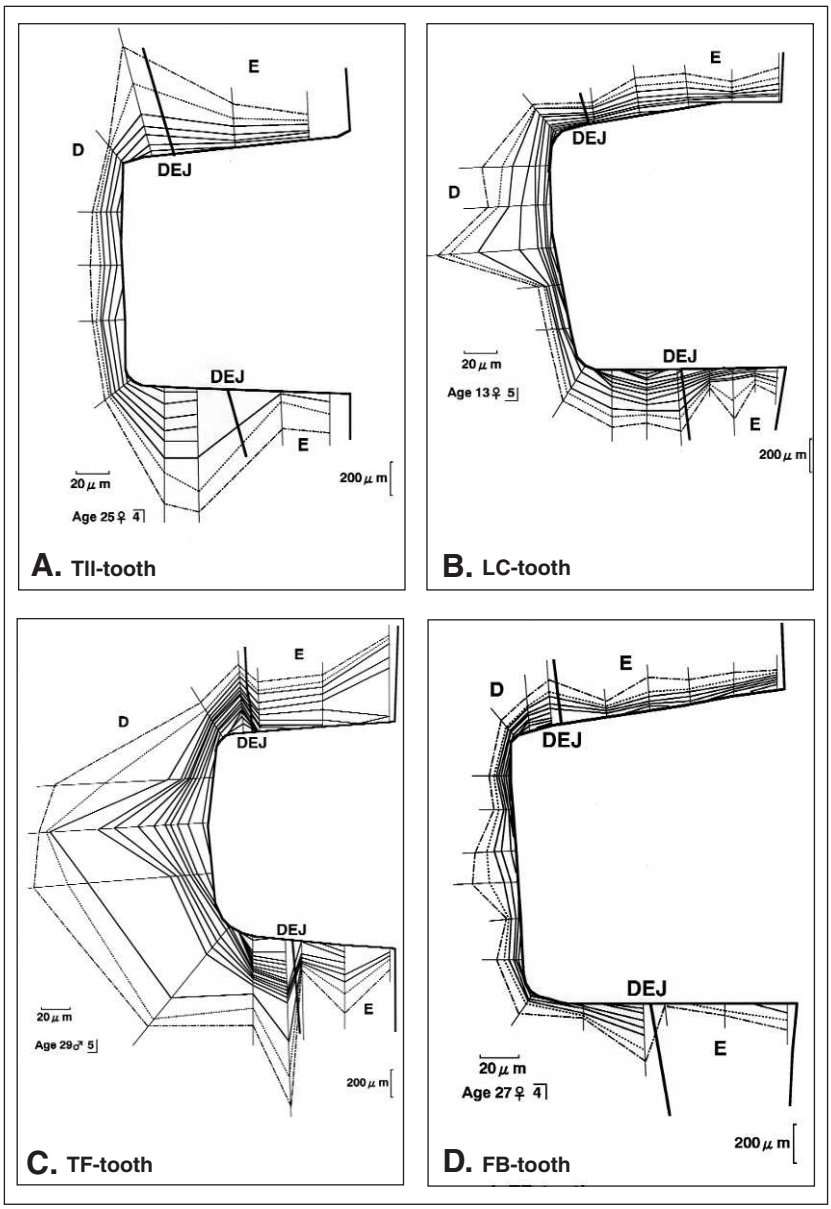

Figure 6. *Map of fluoride distribution around the entire cavity wall after one month for teeth filled with fluoride releasing materials; A) tooth filled with conventional glass ionomer cement, B) tooth filled with light-cured glass ionomer cement, C) tooth filled with light-cured composite resin, D) tooth filled with light-cured bonding agent and non-fluoride releasing, light-cured composite resin. Bold line (▬) shows the cavity wall, fine line (–) shows the places scanned from the surface of the cavity wall towards the inside of the tooth, medium line (—) shows each 1000 fluoride ppm unit, dotted line (·····) shows 500 fluoride ppm, broken line (---) shows base line, which signifies a detection limit.*

D;dentin, E;enamel, DEJ; dentin-enamel junction

RESULTS
(MAPPING OF TEETH WITH FLUORIDE-RELEASING MATERIALS)

Parts of the obtained maps of the TII-, LC-, TF-, and FB-teeth are shown in Figures 6A-D. In Figures 7A-C and 8A-C, the observed figures of surface fluoride concentration and maximal depth of fluoride uptake at the side wall in enamel, and the side and axial walls in dentin are shown. The fluoride-releasing materials in this experiment were divided into three groups according to their application method. TII and LC are cement-form and packed fully into a cavity. TF is resin-form

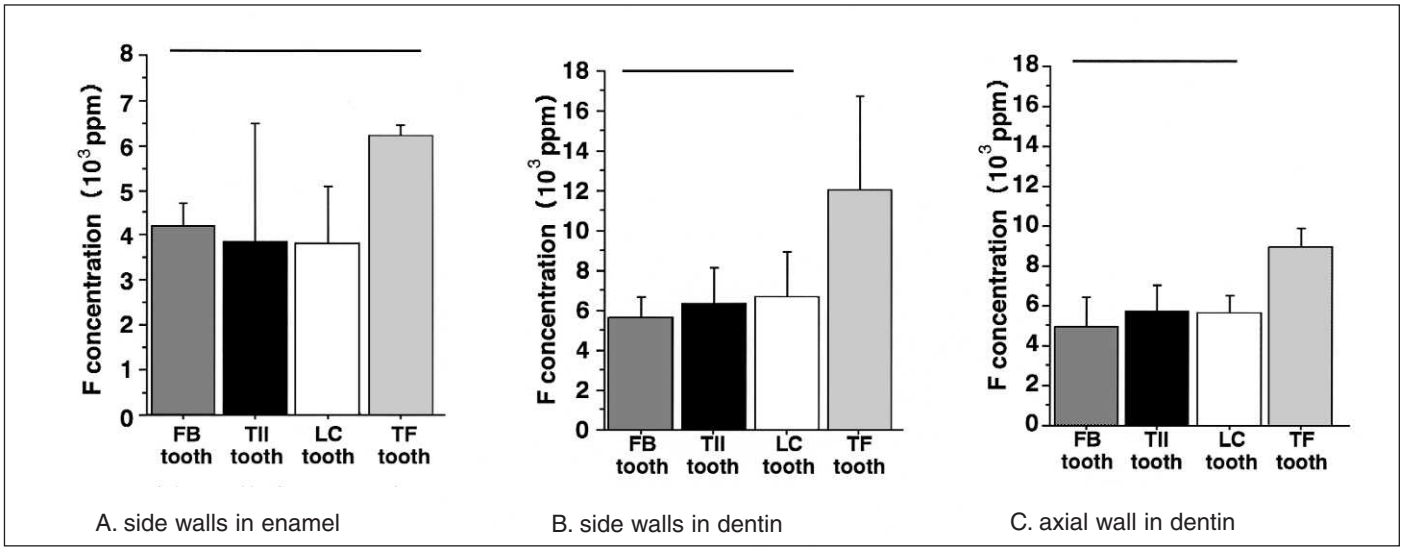

Figure 7. *Surface fluoride concentration by location on cavity wall after one month for teeth filled with fluoride-releasing material. Location: A) side walls in enamel, B) side walls in dentin, C) axial wall in dentin. Horizontal line (—) shows statistically insignificant difference (p<0.05).*

and is also packed fully in a cavity. FB is also resin-form, but is applied by coating it on the surface of a cavity wall. The TII-tooth and the LC-tooth took up fluoride both in enamel and dentin, although the penetration depth in enamel was more shallow than in dentin. Both the TF-tooth and FB-tooth also took up fluoride in enamel and in dentin. The surface fluoride concentrations of the TII-tooth and the LC-tooth at the enamel were nearly the same: 3.9 x 10^3 ppm for the TII-tooth and 3.8 x 10^3ppm for the LC-tooth. At the location of the side wall in dentin the concentration was 6.7 x 10^3 ppm for the TII- and 6.3 x 10^3 ppm for the LC-tooth. Figures of 5.7 x 10^3 ppm for the TII- and 5.6 x 10^3 ppm for the LC-tooth were observed at the axial wall in the dentin. For the TF-tooth, concentrations of 6.2 x 10^3 ppm, 11.8 x 10^3 ppm, and 8.9 x 10^3 ppm were obtained at the enamel surface locations, side wall surface, and axial wall surface of dentin, respectively. As for penetration depth, the TII-tooth and the LC-tooth gave 12 μm and 13 μm in enamel, 44 μm and 45 μm on the side wall of the dentin, and 23 μm and 38 μm at the axial wall of the dentin. The figures for the TF-tooth at enamel, the side wall of dentin, and the axial wall of dentin were 32 μm, 86 μm, and 88 μm, respectively. For the FB-tooth, figures of 11 μm for enamel, 16 μm for the side wall of dentin, and 15 μm for the axial wall of dentin were observed. The above figures are average values obtained under statistical procedure and may not represent the exact fluoride uptake in any one tooth. Yet it can be concluded that the form and application of the fluoride-releasing materials affected the mapping of fluoride uptake; in

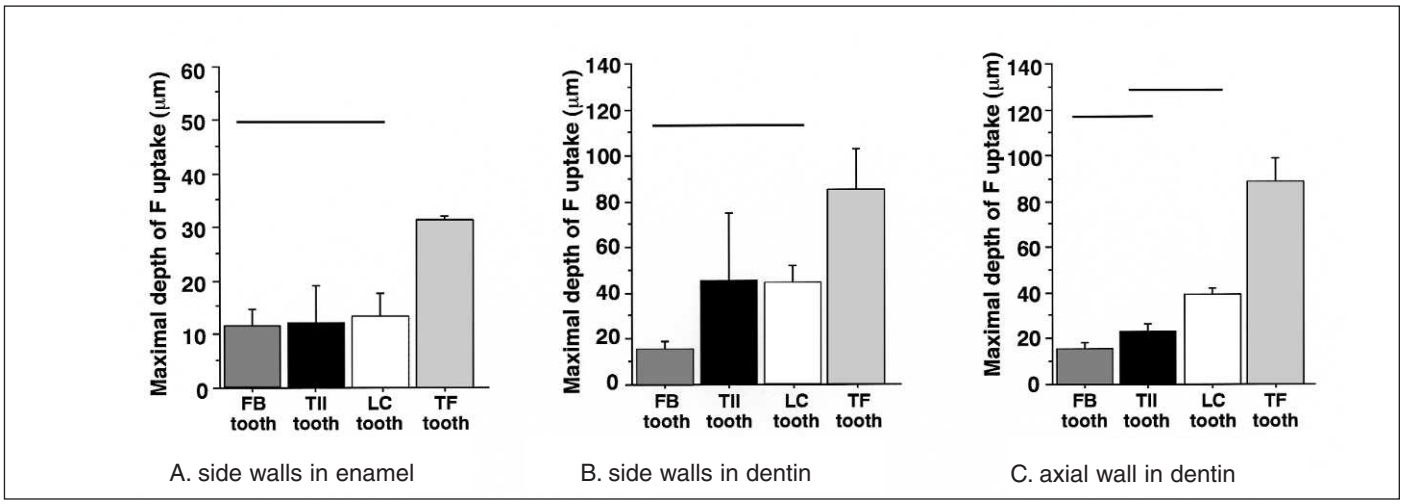

Figure 8. *Maximal depth of fluoride uptake by location on cavity wall after one month for teeth filled with fluoride-releasing materials. Location: A) side walls in enamel, B) side walls in dentin, C) axial wall in dentin. Horizontal line (—) shows statistically insignificant difference (p<0.05).*

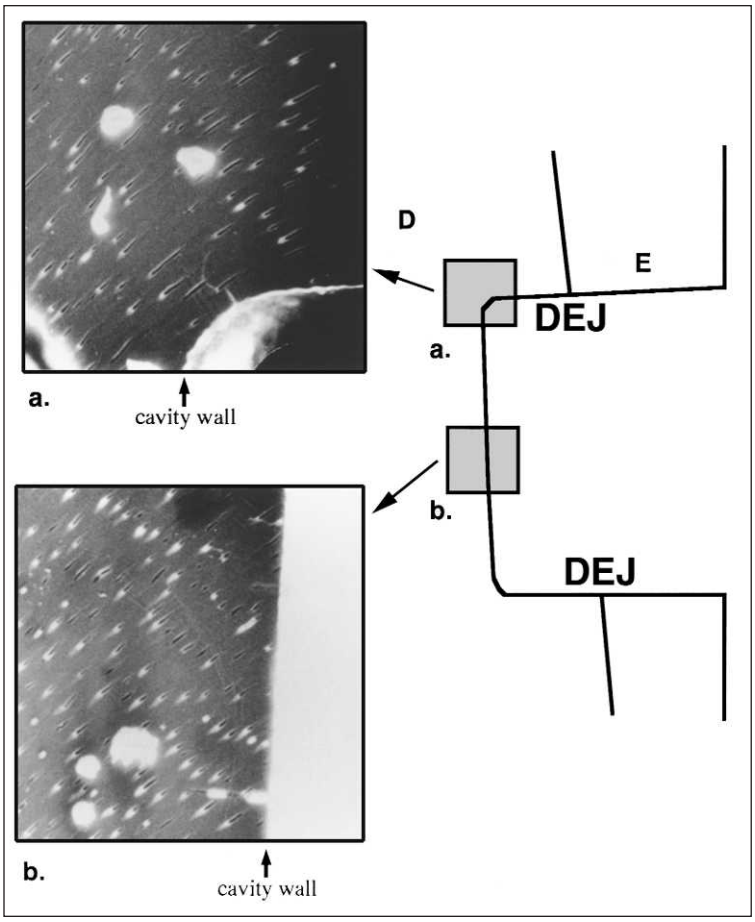

Figure 9. *SEM photographs of dentinal tubule. Locations: a) occuloso-axial line angle, b) axial wall.*

D; dentin, E; enamel, DEJ; dentin-enamel junction

other words, the distribution of fluoride in the tooth. Fluoride penetrated more deeply or smoothly into teeth when using TF as a fluoride-releasing material, though the anisotropic quality of location-dependent uptake was also stronger than the other materials.

DISCUSSION

Mapping of fluoride distribution in a tooth was performed by using a nondestructive method (EPMA). A difference of fluoride uptake between enamel and dentin was observed in all specimens. This was expected because of the difference of the two structures. The difference of the organic microstructure will affect the uptake process of the fluoride. It was also expected that the plain microstructure of enamel would give nearly identical diffusion, despite the differences in form of the fluoride-releasing materials tested. The other, and most important difference, is the strong location dependence of the fluoride uptake in a tooth. All specimens for each fluoride-releasing material group showed the same tendency in fluoride uptake. The results were far from the anticipated result based on Fick's second law of diffusion in a solid material. However, it may be a rather natural result, for Fick's law assumes uniform material structure though teeth have complex secondary and tertiary structural formations. An actual explanation of the location dependence is difficult, involving the fine structure of the material and the observations of the fluoride-penetrating mechanism. Chappell and others (1994) reported the penetration of low-viscosity resin into dentinal tubules, resulting in resin-tag formation, which suggests a second process of fluoride penetration into the dentin. The second process may be as follows: The fluoride in a low-viscosity resin can penetrate deeply into the tooth with the flow of the resin or liquid (in this case NaF-solution) through a dentinal tubule, like a small ship in a canal (ten Cate, Damen & Buijs, 1998). Then the fluoride can diffuse through the wall of the tubule. The diffusion process will thus be faster than that of a solid. In this experiment the process was expected to take place in the NaF-tooth and the TF-tooth, where both fluoride-releasing materials were liquid and low-viscosity resin. Indeed this was the experimental result. Moreover, this assumption was somewhat verified by the SEM photographs of the NaF-tooth in Figures 9a-b. These show the correlation between the state of a tubule and fluoride uptake. A dentinal tubule runs parallel to the cavity wall at the occlusoaxial line angle, while the direction of the dentinal tubule at the axial wall is not parallel, but the mouth of the tubule is open. The location of the open tubule and the direction of the tubule affect the penetration depth of fluoride. If the dentinal tubule paralleled the cavity wall and the tubule mouth had no contact with the fluoride-containing solution, one could expect only slow diffusion from the cavity wall through solid to take place, resulting in a shallow depth of penetration. However, if the tubule were perpendicular to the wall, the solution could infiltrate directly into the tubule and the fluoride ion could diffuse into the solid from the tubule. The TII and LC are cements, so fluoride diffusion may only take place by solid-state diffusion; thus we may expect a slow diffusion process and shallow penetration depth compared with NaF and TF. This expectation was also supported by the experiment. Comparing the results of NaF, TF, TII, and LC, uptake of fluoride into a tooth did not depend upon the quantity of the fluoride release into the saline (Yamamoto, 1995), which was picked up as a model environment. For the FB tooth, the situation was somewhat different from the other four kinds of fluoride-releasing materials. In this material the fluoride supplier is a thin-coated film, though the film is made up of low-viscosity resin. This thin film might restrict the continuous amount of fluoride supply. This resulted in the observed shallow penetration of the fluoride.

In this study, the data were averaged in order to simplify the results at the side walls and axial wall, although the variation in fluoride uptake was not measurement error but was due to the location in a tooth. For this reason, the range of data may be large.

It is clearly shown from the above data that we cannot anticipate the fluoride uptake conditions, such as penetration depth, concentration, or location dependence, from the data of destructive methods (chemical analysis). WDX measurement, though two-dimensional, can give a map of fluoride distribution and permits a full inspection of the fluoride distribution in a tooth. As a whole, it can be concluded that the two-dimensional mapping of fluoride uptake by the EPMA measurement is a valuable method for both fundamental research of caries and also for the evaluation of applied fluoride-releasing materials. Regrettably, one of the most important problems remains unresolved: The experiment was conducted *in vitro* and we do not know if the situation would be the same *in vivo*. Fluoride-uptake research involving *in vivo* conditions is now underway.

CONCLUSION

1. Two-dimensional maps of fluoride distributed around cavity walls were drawn using WDX line analysis.

2. It was confirmed that the amount of fluoride uptake around the cavity wall varied depending on the location.

3. The fluoride uptake in dentin was larger than that in enamel.

4. The amount of fluoride uptake from fluoride-releasing resin was larger than that from glass-ionomer cement regardless of the hardening process.

5. Two-dimensional mapping of fluoride uptake from fluoride-releasing materials could provide useful information when developing new fluoride-releasing materials.

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Effectiveness of Polymerization in Composite Restoratives Claiming Bulk Placement: Impact of Cavity Depth and Exposure Time

AUJ Yap

Clinical Relevance

The composite restoratives evaluated should not be placed in increments greater than 2 mm in order to obtain uniform and maximum polymerization. The findings did not support the manufacturer's claim of "bulk placement."

SUMMARY

The impact of variation of cavity depth and light-source exposure time upon the effectiveness of polymerization of two "bulk placement" composite restoratives was assessed indirectly using hardness testing. The current investigation used a digital microhardness tester to evaluate hardness gradient between top and bottom surfaces of composite specimens of various depths after different light-exposure times. The results showed that the effectiveness of polymerization decreased significantly with increased cavity depth regardless of exposure time. Increased exposure time increased the extent of polymerization at cavity depths of 3 to 4 mm. Increments of the composites evaluated should be no greater than 2 mm to provide uniform and maximum polymerization.

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INTRODUCTION

Light-activated composite resin restoratives have revolutionized clinical dentistry by maximizing working time and minimizing setting time. Since their introduction in the late 1970s, there has been a rapid increase in the number of composite formulations and range of products available. One of the limitations of these materials is that a hard-top surface is not an indication of adequate polymerization throughout the depth of the restoration (Pilo & Cardash, 1992). The effectiveness of polymerization or depth of cure of light-activated composites is vital, not only to ensure optimum physiomechanical properties (Asmussen, 1982a), but also to ensure that clinical problems do not arise because of the cytotoxicity of inadequately polymerized material (Caughman & others, 1991). The effectiveness of polymerization is dependent not only on the chemistry of the material and concentration of initiator, but also upon the filler particle type, size, and

quantity. In addition, polymerization is dependent on the effectiveness of the radiation sources, including spectral distribution, intensity, exposure time, and alignment of the light-tip guide (Harrington, Wilson & Shortall, 1996).

The effectiveness of polymerization may be assessed directly or indirectly. Indirect methods have included scraping (Cook, 1980), visual (Murray, Yates & Newman, 1981), and surface hardness (Asmussen, 1982b). Incremental surface hardness has been used in many studies because surface hardness has been shown to be an indicator of the degree of polymerization (Asmussen, 1982b). Direct methods that assess degree of conversion, such as laser Raman spectroscopy (Louden & Roberts, 1983) and infrared spectroscopy (Asmussen, 1982a), have not been accepted for routine use because the methods are complex, expensive, and time consuming (Rueggeberg & Craig, 1988). DeWald and Ferracane (1987) compared four commonly used methods for evaluating depth of cure in light-activated composites. They found that visual and scraping methods correlated well, but severely overestimated depth of cure as compared with hardness testing or degree of conversion. Degree of conversion appeared to be the most sensitive test for depth of cure. A good correlation between the results of hardness and infrared spectroscopy experiments using Knoop hardness testing was also reported. Hardness testing appears to be the most popular method for investigating factors that influence effectiveness of polymerization because of the relative simplicity of the method.

The purpose of this investigation was to study the impact via hardness testing of variation of cavity depth and light-source exposure time on the effectiveness of polymerization of two "bulk placement" composite restoratives. A secondary objective was to validate claims by the manufacturers that their restoratives were formulated to be sufficiently cured to depths of 4 to 5 mm with 40 seconds of visible-light exposure.

METHODS AND MATERIALS

The two bulk placement composite restoratives chosen were Ariston pHc (Vivadent, Schaan, Liechtenstein) and Surefil (Dentsply Inc, Milford, DE 19963) of shade A2. Both composites are based upon BIS-GMA resin with urethane dimethacrylate or urethane modification. The filler components of both materials consist of barium aluminofluorosilicate glasses and silica. Ariston, in addition, contains ytterbiumtrifluoride fillers and alkaline glass fillers that release fluoride, calcium, and hydroxyl ions. The filler loading in weight percent is about 79% for Ariston and 82% for Surefil.

The composite restoratives under test were placed in translucent plastic molds with cylindrical cavities 2 to 4 mm deep and 5 mm in diameter. A black backing was used beneath the molds, and the material placed in the mold cavity was confined between opposing cellulose acetate strips (Hawe-Neos Dental, Bioggio, Switzerland). A glass slide (1 mm thick) was then placed on the molds, and excess material was extruded out by applying pressure. The materials were then irradiated from the top surface through the glass slide for 40 seconds, as recommended by the manufacturers, with a Spectrum Curing Light (Dentsply Inc). The intensity of the light source was checked with a radiometer (Cure Rite; EFOS Inc, Ontario, Canada) before the start of each experimental session. The mean output was 421.33 ± 1.5 mW/cm^2 and output was not affected by illumination through the glass slide and acetate strip. Immediately after light activation, the acetate strips were discarded, and the specimens, in their molds, were positioned centrally beneath the indentor of a digital microhardness tester (FM7; Future-Tech Corp, Tokyo, Japan) to assess Knoop's hardness (KHN) of the top and bottom surfaces. A 500 gf load was applied through the indentor with a dwell time of 15 seconds. Three specimens were made for each material and cavity-depth combination.

KHN readings were recorded after 40 seconds of light irradiation. The specimens were then further irradiated in 20-second increments up to 120 seconds from the top surface through the glass slide. Hardness readings at the top and bottom surfaces were taken at each time increment, and the mean KHN and the hardness ratio were then calculated and tabulated. The hardness ratio was calculated using the following formula: Hardness ratio = KHN of bottom surface/KHN of top surface.

If polymerization was effective (ie, maximum cure of the specimen was achieved) for the depth concerned, the ideal ratio should be 1:1, as the hardness of the bottom surface should be similar to that of the top surface. The data obtained were subjected to one-way ANOVA and Scheffé's post hoc test at a significance level of 0.05 with cavity depth and exposure time as independent variables.

RESULTS

The mean KHN and standard deviation for Ariston and Surefil for the different thicknesses and exposure times are shown in Tables 1 and 2. Figures 1 and 2 reflect the change in mean KHN of the top and bottom surfaces of Ariston and Surefil with increased exposure times, respectively. The hardness ratios of the restoratives at the different cavity depths are shown in Figures 3 and 4. The results of the statistical analysis are summarized in Tables 3 and 4.

For Ariston, there was no significant difference in KHN among the different depths at the top surfaces at the different exposure times (ie, 40 to 120 seconds). At

Table 1: *Mean KHN of Ariston pHc for the different thickness and exposure time.*

Exposure Time	2 mm			3 mm			4 mm		
	Top	Bottom	Hardness Ratio	Top	Bottom	Hardness Ratio	Top	Bottom	Hardness Ratio
40 secs	29.07 (1.76)	26.30 (1.55)	0.90 (0.01)	26.10 (1.80)	13.23 (0.15)	0.51 (0.03)	30.10 (1.47)	9.17 (2.02)	0.30 (0.05)
60 secs	30.87 (1.60)	27.77 (1.17)	0.90 (0.01)	28.40 (0.57)	17.20 (0.75)	0.61 (0.03)	30.83 (1.90)	11.37 (1.60)	0.37 (0.04)
80 secs	32.23 (1.65)	29.83 (2.24)	0.93 (0.02)	30.37 (1.45)	19.77 (0.21)	0.65 (0.02)	32.30 (1.25)	12.27 (1.15)	0.38 (0.03)
100 secs	33.64 (1.64)	30.87 (1.78)	0.93 (0.02)	33.40 (1.77)	21.80 (1.25)	0.67 (0.01)	33.20 (1.55)	14.97 (1.95)	0.45 (0.05)
120 secs	33.83 (1.19)	31.60 (1.30)	0.93 (0.02)	33.87 (1.42)	24.10 (1.34)	0.71 (0.03	34.07 (1.27)	16.70 (0.61)	0.49 (0.01)

Table 2: *Mean KHN of Surefil for the different thickness and exposure time.*

Exposure Time	2 mm			3 mm			4 mm		
	Top	Bottom	Hardness Ratio	Top	Bottom	Hardness Ratio	Top	Bottom	Hardness Ratio
40 secs	38.80 (1.90)	37.63 (0.32)	0.97 (0.04)	39.97 (1.97)	21.37 (10.80)	0.53 (0.15)	39.80 (3.36)	14.57 (2.34)	0.37 (0.05)
60 secs	46.23 (1.25)	44.30 (2.81)	0.96 (0.04)	49.40 (5.73)	30.43 (1.41)	0.62 (0.10)	44.33 (3.46)	16.73 (1.05)	0.38 (0.04)
80 secs	46.40 (1.01)	44.0 (2.21)	0.95 (0.03)	50.90 (5.65)	37.43 (3.25)	0.74 (0.13)	45.20 (3.50)	21.06 (3.38)	0.47 (0.05)
100 secs	53.77 (0.11)	53.43 (0.25)	0.99 (0.01)	53.17 (3.51)	38.57 (1.89	0.73 (0.05)	47.53 (2.85)	23.57 (2.37)	0.50 (0.03)
120 secs	55.43 (3.35)	53.67 (1.76)	0.97 (0.02)	55.40 (2.46)	40.87 (4.17)	0.74 (0.11)	48.17 (3.21)	25.07 (2.23)	0.52 (0.01)

the bottom surfaces, however, the KHN of specimens with cavity depths of 2 mm were significantly harder than that for the 3 and 4 mm depths for all exposure times. The bottom surfaces of the 3 mm specimens were in turn significantly harder than those of the 4 mm specimens. On the top surface, increase in exposure time did not result in a harder surface for the 2 and 4 mm specimens. A significant difference in hardness, however, was found for the 3 mm specimens. At the bottom surfaces, increased exposure time resulted in a general increase in hardness (Figure 1). Significant differences among the different exposure times at the different cavity depths are shown in Table 3.

The hardness ratio for 2 mm-deep Ariston specimens was significantly greater than that for 3 and 4 mm specimens for all exposure times. The 3 mm-deep specimens in turn had greater hardness ratios than the 4 mm specimens. There is, therefore, a significant decrease in the effectiveness of polymerization with increased cavity depths. A significant increase of the hardness ratio, and thus effectiveness of polymerization, was noted with increased exposure times for the 3 and 4 mm specimens (Figure 3). For the 3 mm specimens, the hardness after 40 seconds of light activation was significantly less than that after 60 to 120 seconds. Hardness after 60 seconds of irradiation was significantly lower than that after 120 seconds. For the 4 mm-deep specimens, the hardness ratio after 40 seconds of irradiation was significantly less than that after 100 and 120 seconds. For the 2 mm specimens, an increase in hardness ratio was not significant with increased exposure time.

For Surefil, there was also no significant difference in hardness of the top surfaces among the different cavity depths at the different exposure times. For 60, 100, and 120 seconds of exposure, the hardness at the bottom surfaces of the 2 mm specimens was significantly greater than that for specimens that were 3 and 4 mm deep. The 3 mm specimens had significantly harder bottom surfaces than those of the 4 mm specimens. For 40 seconds of irradiation, only the 2 mm specimens had significantly harder bottom surfaces than the 4 mm specimens. After 80 seconds of exposure time, both the 2 and 3 mm specimens had significantly higher KHN

Figure 1: *Mean KHN of Ariston pHc with increase light exposure time.*

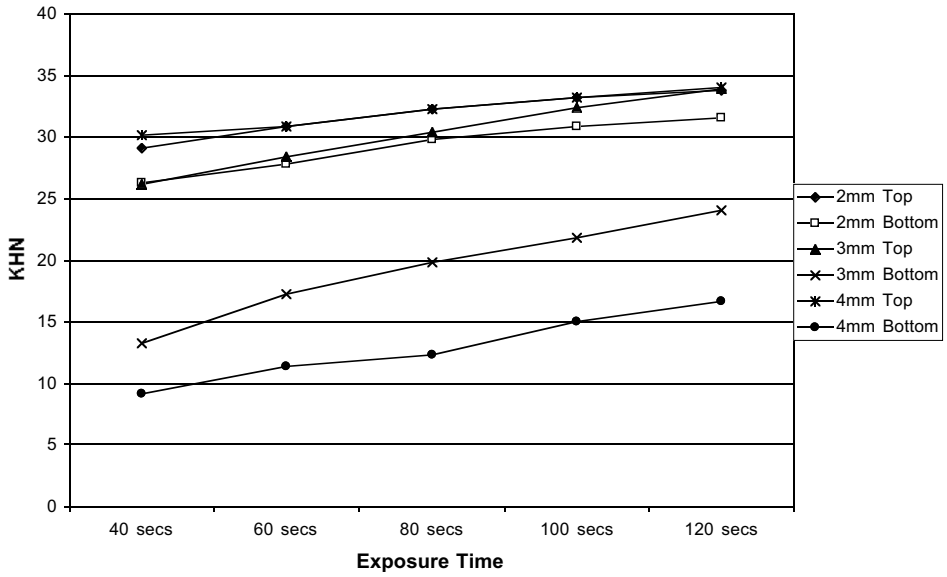
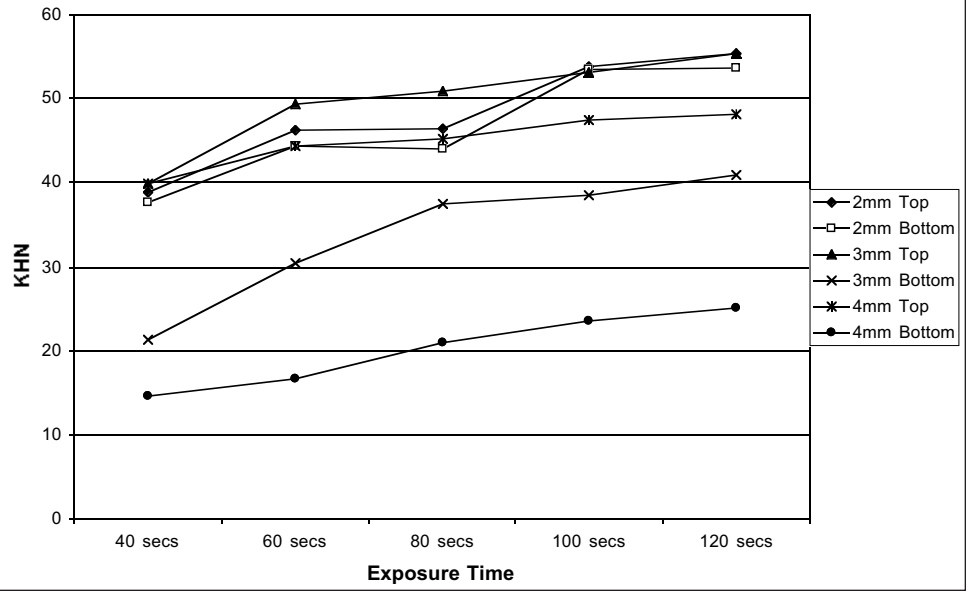


Figure 2: *Mean KHN of Surefil with increase light exposure time.*



at the bottom surface than the 4 mm specimens. With an increase in exposure time, there was an increase in hardness of the top surfaces of the 2 and 3 mm-deep specimens. No significant difference in hardness on the top surface among the various exposure times was found for 4 mm specimens. At the bottom surfaces, increased exposure times resulted in significantly harder surfaces, as reflected in Figure 2 and Table 4.

With the exception of specimens light activated for 80 seconds, Surefil had a similar pattern of statistical significance for hardness ratio to Ariston for the various exposure times. For Surefil, light activated for 80 seconds, the hardness ratio of the 2 and 3 mm specimens were significantly greater than that of the 4 mm specimens. Increase in cavity depths again decreased effectiveness of polymerization for all exposure times. The hardness ratio of the 2 mm specimens remained about the same but increased with increased exposure time for the 3 and 4 mm specimens (Figure 4). No significant difference in hardness ratio was found among the different exposure times for the 2 and 3 mm-deep specimens (Table 4). For the 4 mm specimens, however, the hardness ratio after 40 seconds of irradiation was significantly less than that for 100 and 120 seconds of exposure. The hardness ratio after 60 seconds of exposure was significantly lower than that for 120 seconds.

DISCUSSION

Bulk placement of composites may be anticipated in deep Class I and Class II cavities and the restoration of endodontically-treated teeth if they are to used as an amalgam substitute. It is, however, difficult for visible light to penetrate thick layers of composites, particularly those with darker shades. Filler particles, colorants, and pores scatter visible light and reduce curing efficacy. The actual intensity of light for curing is dependent on the intensity produced by the curing unit, the distance of the light-curing tip from the surface of the material, and internal light scattering within the composite (Bayne, Heymann & Swift, 1994). One possible solution to the curing-depth problem has been the Argon laser-curing system, which can provide curing up to 11 mm (Blankenau & others, 1991; Bayne & others, 1994), and the other is a change in composite formulation. The manufacturers of the composites evaluated claimed that their products were formulated to be placed in bulk increments of 4 to 5 mm with a single 40-second visible-light exposure. Bulk placement of composites may also generate high internal stresses in the material and loss of marginal integrity, as the larger

Figure 3: *Hardness ratio for Ariston pHc at the different cavity depths.*

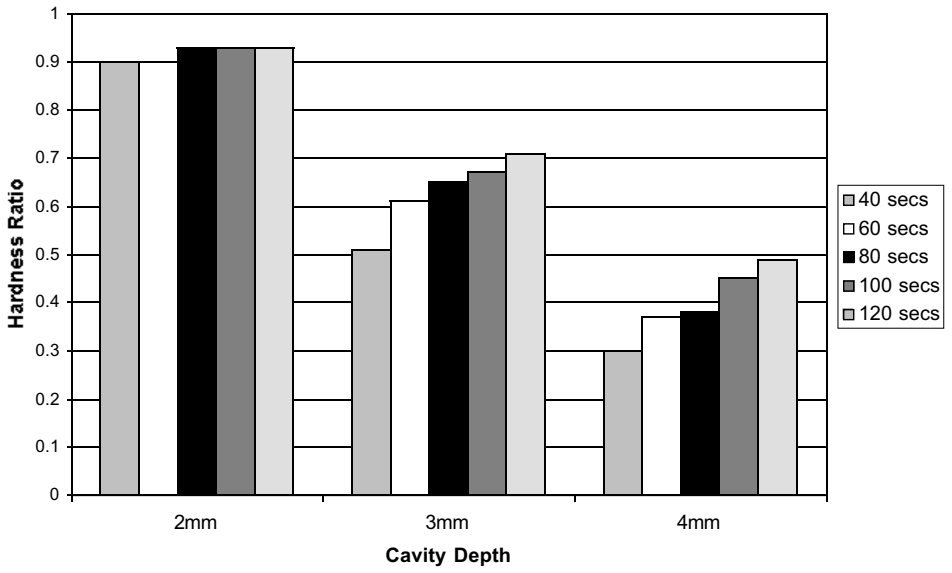
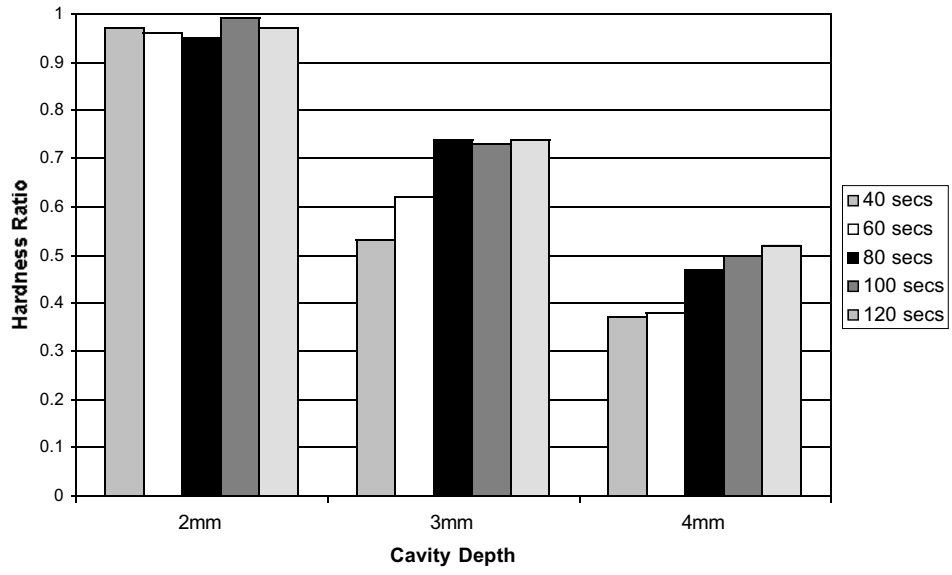


Figure 4: *Hardness ratio for Surefil at the different cavity depths.*



the volume of composite to be polymerized, the larger will be the polymerization shrinkage (Roulet, 1997).

The degree to which light-activated composites polymerize is proportional to the amount of light to which they are exposed (Rueggeberg, Caughman & Curtis, 1994). At the upper surface of the composites, where no overlying composites interfere with light transmission, it has been found that even a curing source with relatively low intensity can cure the resin matrix to an extent almost equal to that when the high-intensity lights are used (Rueggeberg & Jordan, 1993). In the ideal situation, the degree of polymerization of the composite should be the same throughout its depth, and the hardness ratio should be equal to 1:1 or very close to it. As light passes through the bulk of the composite, the light intensity is greatly reduced due to light scattering, thus decreasing the effectiveness of polymerization (Ruyter & Øysæd, 1982). This scattering of light accounts for the minor differences in hardness between the top and bottom surfaces of the composites evaluated when specimens were 2 mm deep. It has been suggested that the top-to-bottom hardness gradient should not exceed 10-20% (ie, hardness ratio should be greater than 0.8) for adequately polymerized photo-activated resin composite (Yearn, 1985; Pilo & Cardash, 1992). Watts, Amer, and Combe (1984) considered that the depth at which a 20% reduction had taken place in maximum hardness to be a realistic measure of depth of cure. Ruyter and Øysæd (1982) suggested that the scattering was greatest when the filler-particle size was approximately one-half the wavelength of the activating light. The increasing trend for manufacturers to achieve finer particle-size distribution in commercial composites makes this an important consideration.

At depths greater than 2 mm, poor polymerization of composite resulted, for polymerization was very susceptible to changes in light intensity and exposure duration (Rueggeberg & others, 1994). The light-source intensity was thus checked with a radiometer before each experimental session. With the new bulk placement materials, manufacturers have claimed that their high-density composites will be sufficiently polymerized up to a depth of 4 to 5 mm with a single 40-second exposure using a curing-light device of output greater than 300 mW/cm^2 (Dentsply, 1998; Vivadent, 1998). Since Barron, Rueggeberg, and Schuster (1992) demonstrated an inverse relationship between inorganic filler loading and monomer conversion, the claims by these manufacturers must be validated. Based on a hardness

Table 3: *Intergroup comparison of KHN and hardness ratio for Ariston pHc.*

EXPOSURE TIME	Top Surface	Bottom Surface	Hardness Ratio
40 secs	NS	2 mm > 3 mm, 4 mm; 3 mm > 4 mm	2 mm > 3 mm, 4 mm; 3 mm > 4 mm
60 secs	NS	As above	As above
80 secs	NS	As above	As above
100 secs	NS	As above	As above
120 secs	NS	As above	As above
CAVITY DEPTH	Top Surface	Bottom Surface	Hardness Ratio
2 mm	NS	40 secs < 120 secs	NS
3 mm	40 secs < 100, 120 secs; 60 secs < 120 secs	40 secs < 100, 120 secs; 60 secs < 100, 120 secs; 80 secs < 120 secs	40 secs < 60, 80, 100, 120 secs; 60 secs < 120 secs
4 mm	NS	40 secs < 100, 120 secs; 60 secs < 120 secs	40 secs < 100, 120 secs

NS denotes no statistical significance.

Table 4: *Intergroup comparison of KHN and hardness ratio for Surefil.*

EXPOSURE TIME	Top Surface	Bottom Surface	Hardness Ratio
40 secs	NS	2 mm > 4 mm;	2 mm > 3 mm, 4 mm; 3 mm > 4 mm
60 secs	NS	2 mm> 3 mm, 4 mm; 3 mm > 4 mm	2 mm > 3 mm, 4 mm; 3 mm > 4 mm
80 secs	NS	2 mm, 3 mm > 4mm	2 mm > 3 mm, 4 mm
100 secs	NS	2 mm > 3 mm, 4 mm; 3 mm > 4 mm	2 mm > 3 mm, 4 mm; 3 mm > 4 mm
120 secs	NS	2 mm > 3mm, 4 mm; 3 mm > 4 mm	2 mm > 3 mm, 4 mm; 3 mm > 4 mm
CAVITY DEPTH	Top Surface	Bottom Surface	Hardness Ratio
2 mm	40 secs < 60, 80, 100, 120 secs; 60, 80 secs < 100, 120 secs	40 secs < 60, 80, 100, 120 secs; 60, 80 secs < 100, 120 secs	NS
3 mm	40 secs < 100, 120 secs	40 secs < 100, 120 secs	NS
4 mm	NS	40 secs < 100, 120 secs; 60 secs < 120 secs	40 secs < 100, 120 secs; 60 secs < 120 secs

NS denotes no statistical significance.

ratio of 0.8 as the difference that can be tolerated between hardness of top and bottom surfaces, polymerization of both Ariston and Surefil was only effective up to 2 mm when exposure time was 40 seconds. The hardness ratio for the 2 mm specimens of both composites was, in general, significantly greater than that of the 3 and 4 mm specimens. The hardness ratio of the 3 mm specimens was in turn greater than that for the 4 mm specimens. The effectiveness of polymerization thus decreased significantly with an increase in cavity depth. Mean hardness ratio for a single 40-second exposure was 0.90 and 0.97 for Ariston and Surefil,

respectively. The hardness ratio after a 40-second exposure time dropped by 43.3% to 0.51 and 66.6% to 0.30 for the 3 and 4 mm-deep Ariston specimens. The hardness ratio for Surefil dropped by 45.4% to 0.53 for the 3 mm specimens and 61.9% to 0.37 for the 4 mm specimens for the same exposure time. Polymerization using a 40-second exposure time is thus inadequate at depths of 3 and 4 mm, as results were negative even at 4 mm, investigation based on a 5 mm cavity depth was not pursued.

Since a 40-second exposure time, as suggested by the manufacturers, did not provide adequate polymerization, light-exposure time was increased up to 120 seconds to study the effects of exposure time on effectiveness of polymerization at various depths. In general, an increase in hardness was noted with increased exposure times for both composites at the different depths. This finding suggested that a greater extent of polymerization is obtained with increased exposure times. It is, however, not practical to use extended exposure times for all cavity depths, as the minor increase in hardness and indirect polymerization does not justify the investment of the additional clinical time. For Ariston and Surefil at 2 mm depths, increased exposure did not result in a significant increase in the hardness ratio, although significant differences were noted at the top surface of Surefil and bottom surfaces of both composites with increased exposure times. Increased hardness of the top surface with increased exposure time, if present, was accompanied by an increase in hardness of the bottom surface. This confirmed that polymerization was effective throughout the depth of the cavity.

When the cavity depth is 3 to 4 mm, Ariston should be irradiated for a minimal exposure time of 100 seconds. This exposure time was the smallest at which a significance in hardness ratio was noted when compared to a single 40-second exposure. A 120-second exposure time, however, is best to ensure maximum polymerization. For Surefil, there was no significant difference in hardness ratio for the 3 mm specimens among the different curing times. Any increase in hardness of the top surface was accompanied by an increase in hardness of the bottom surface. The hardness ratios of the 3 mm specimens were, however, significantly lower than those of the 2 mm specimens but greater than those for the 4 mm specimens. When cavity depth was 4 mm, a minimal exposure time of 100 seconds is also recommended. The best cure was again achieved with 120 seconds of light irradiation.

Despite the increased effectiveness of polymerization with increased exposure time for the 3 to 4 mm-deep specimens, it is important to note that the hardness ratio was only 0.71 to 0.74 with a 120-second exposure time. This is still below the tolerable hardness ratio of 0.8. Because of clinical factors. such as the accessibility of the light source, the direction of light and intervening tooth tissue, the effectiveness of light polymerization obtained clinically may be less than achieved under the ideal laboratory conditions of this experiment. In addition, greater depths of cure had been reported for measurements made in plastic molds than those made in metal molds (Yearn, 1985). Increments of the new bulk placement composites evaluated should, therefore, be no greater than 2 mm to provide uniform and maximum polymerization despite manufacturers' claims. The polymerization shrinkage and the degree of conversion associated with the bulk placement of these composites warrants further investigation.

CONCLUSIONS

1. Increments of the composites evaluated should be no greater than 2 mm to obtain uniform and maximum cure.

2. Increased cavity depth resulted in a significant decrease in the effectiveness of polymerization for all exposure times.

3. Increased exposure time resulted in an increase in hardness ratio, and indirectly, effectiveness of polymerization at cavity depths of 3 to 4 mm.

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Literature Review

Bonding of Amalgam Restorations: Existing Knowledge and Future Prospects

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Clinical Relevance

Numerous *in vitro* studies have demonstrated potential advantages with bonded amalgam, including improved retention, decreased microleakage, and possible tooth reinforcement. However, the few *in vivo* studies available show little advantage for bonding in traditional amalgam preparations with mechanical undercuts.

SUMMARY

A number of laboratory and clinical studies over the last 15 years have explored the potential advantages of bonding amalgam to tooth surfaces. Bond strengths have been reported to range from 2 to 20 MPa, with higher bond strengths reported for filled adhesives. Most studies agree that the use of bonding results in a considerable reduction of microleakage, when compared with copal varnish or no lining. The use of bonding provides retention in vitro **that is equivalent to, or better than, the use of mechanical undercuts. Most studies on strength of restored teeth report an improvement in resistance to fracture or cuspal flexing as measured by strain gauges. Penetration of secondary caries along the interface has been found to be inhibited by bonding. The mode of failure of bonded amalgams has generally been reported to be mixed, but predominantly between the resin and amalgam.** In vitro **studies have reported one potential problem in the incorporation of resin into amalgam, which may cause a decrease in strength of the restoration.**

The clinical studies of bonded amalgams that have been published to date are of short duration, indicating that when traditional preparations are used, no problems are seen with bonding, but also no advantages, as measured by clinical assessment criteria. However, some studies show that bonded amalgam may be useful for procedures where non-bonded amalgams would be expected to be lost, namely in preparations with little, if any, mechanical retention.

It was concluded that, while there are various in vitro **studies demonstrating that bonded amalgams have advantages of improved retention and tooth reinforcement and decreased marginal microleakage and secondary caries, the operative technique is more complicated and there are few advantages yet evident from clinical studies in conventional preparations having mechanical retentive features. However, there is evidence accruing from clinical studies**

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that bonding of amalgam can be favorably used to extend the range of usage of amalgam to non-retentive conservative preparations, and toward the other extreme, as an adjunct to other forms of retention in large compound restorations.

INTRODUCTION

The development of bonded amalgams was brought about by an attempt to determine if the advantages of bonding resin composites could be utilized to overcome some of the inherent limitations of traditional amalgam restorations. Studies have been and are still being carried out to investigate bonded amalgams.

IN VITRO STUDIES

Retention

A goal of amalgam bonding is to create a strong, durable bond between tooth structure and amalgam. Traditional amalgam restorations are retained by preparation features that incorporate parallel or undercut walls, dovetails, box forms, and retention grooves. Such preparations often require removal of healthy tooth structure.

Staninec (1989) tested the retention of bonded amalgam restorations in dental preparations without undercuts in comparison to non-bonded amalgams retained with undercuts. He found that the bonded amalgam restorations showed higher resistance to dislodgment. Therefore, at least in the short-term, bonding provides much greater retention than, for example, grooves or dovetails. He also found that the bonding technique helped conserve tooth structure by requiring removal of less sound tooth tissue.

Charlton, Moore, and Swartz (1992) reported that restorations placed in preparations lined with Amalgambond (Parkell, Farmington, NY 11735) or Panavia EX (Kuraray, Osaka, Japan) exhibited significantly greater retention than restorations placed in unlined preparations or preparations lined with Prisma Universal Bond 2 (LD Caulk/Dentsply, Milford, DE 19963) or Copalite (Cooley & Cooley, Ltd, Houston, TX 77041). Eakle and others (1994) studied the retention of amalgam and gallium restorations bonded with Amalgambond, Panavia, and All-Bond with Liner F (Bisco Inc, Schaumburg, IL 60193). They found that adhesive retention was at least equal to, and in some combinations better than, mechanical undercuts.

Evidence of improved retention from the bonding of amalgam restorations is also emerging from several clinical studies. The scope of these studies ranges from traditional preparations and those with no deliberate retention (Ruzickova & others, 1997; Setcos, Staninec & Wilson, 1998), unprepared pits and fissures (Staninec & others, 1998), to extensive restorations compared with pin-retained restorations (Burgess, Alvarez & Summitt, 1997. (See also the section, Clinical Studies).

Bond Strengths

The strength of the composite-to-enamel bond (20-25 MPa), is considered to be the best tooth-restorative bond presently available (Barkmeier, Shaffer & Gwinnett, 1986; Swift & Cloe, 1993). Retief, Mandras, and Russell (1994) estimated that shear bond strength of 21-24 MPa would eliminate microleakage at the dentin-material interface. This bond strength was considered to be sufficient to counteract dimensional changes of composites, namely polymerization shrinkage and temperature-related changes. A similar determination has not been made for amalgams. However, since they are more dimensionally stable, it is likely that the bond strength requirement may be less than 20 MPa. Triolo and Swift (1992) tested the shear bond strengths of nine so-called third-generation dentinal bonding systems with amalgam and found that Amalgambond and All-Bond had the strongest bonds to dentin, 23.3 ± 5.7 and 19.3 ± 5.6 Mpa, respectively. Clearfil Photo Bond (Kuraray) and Prisma Universal Bond 3 (LD Caulk/Dentsply) had intermediate bond strengths, and Gluma (Bayer/Miles Dental, South Bend, IN 46614), Power Bond (Cosmedent, Chicago, IL 60640), Scotchbond 2 (3M Dental Products, St Paul, MN 55144), Tenure (Den-Mat, Santa Maria, CA 93456), and XR Bond (Kerr/Sybron, Orange, CA 92867) all had mean shear bond strengths of less than 8.0 MPa.

However, according to other studies, the strength of the amalgam bonded to tooth has been found to range from 3 to 10 MPa (McComb, Brown & Forman, 1995; Bagley, Wakefield & Robbins, 1994; Barkmeier & others, 1994). Kawakami & others (1991) compared the bond strengths of resinous adhesives used to bond amalgam to dentin. They concluded that All-Bond with Liner-F and Dispersalloy (Dentsply/Caulk, Milford DE 19963-0359) had significantly higher bond strengths than other amalgam-adhesive combinations. Bond strengths of 10-11 MPa were obtained. Although a recent study by Silva and others (1993) reported a shear bond strength of 13.0 MPa for amalgam bonded to dentin with All-Bond 2, the bond strengths of amalgam can be 10 MPa lower than those of composite resin.

Filler Adhesive Liners

Cobb and Diefenderfer (1997) conducted a study to determine the shear bond strength of a spherical alloy (Tytin; Kerr, Romulus, MI 48174) to dentin using several current adhesive resin systems: One-Step and Resinomer (OSR) (Bisco) All-Bond 2 and Resinomer (ABR) (Bisco), Amalgambond Plus with HPA powder (APHPA) (Parkell), Fuji Duet (FD) (GC, Tokyo, Japan), OptiBond FL (OFL), and OptiBond FL with 3A/3B adhesive (OFL3A3B) (Kerr). They found that APHPA produced significantly higher amalgam-to-dentin shear bond strengths than did OFL, OSR, or FD. Bond strengths of amalgam to dentin for APHPA, ABR, and OFL3A3B were significantly greater than FD.

In a similar study, Kline and Boyer (1996) compared bonding of amalgam-to-dentin using several filled adhesives: All-Bond 2 (AB2-unfilled adhesive control); All-Bond 2 + Resinomer; One-Step + Resinomer; OptiBond dual cure; and Amalgambond Plus/HPA (AMP). Etchant, primer, and adhesive were used according to the manufacturer's instructions in each case. It was found that the trend was higher shear bond strengths for bonding amalgam to dentin using filled adhesives, although only one filled adhesive, AMP, was statistically stronger than the unfilled control, AB2. These results are in agreement with Diefenderfer and Reinhardt (1997), who compared the shear bond strengths of amalgam to dentin using two amalgam alloys (Tytin and Dispersalloy) and five adhesive resin systems; filled resin systems (Amalgambond Plus with HPA Powder, OptiBond, and Resinomer) and the unfilled systems (All-Bond 2, Amalgambond Plus). They found that the filled resins tended to produce higher bond strengths (7-14 MPa) than the unfilled resins (4-8 MPa). Similarly, for four of the five resins, a spherical alloy (Tytin) produced higher bond strengths than an admixed alloy (Dispersalloy).

Kim and Lim (1997) evaluated the shear bond strength of amalgam to dentin treated with various bonding agents and examined the mode of failure after the bond test. Four kinds of dental amalgams (two conventional amalgams and two high copper amalgams) and six dentin bonding agents: All-Bond 2, Bond-It (Jeneric/Pentron, Wallingford, CT 06492), Superbond C&B (Sun Medical, Kyoto, Japan), Panavia Ex, Probond, and Bondix (Yamahachi Dental Mfg, Aichi, Japan) were used in this study. Copalite (Cooley & Cooley, Ltd, Houston, TX 77041) varnish was used for the control group. The bonding agents that included NTG-GMA showed a low shear bond strength (3.4-6.2 MPa). The bonding agents that included 4-META showed a high shear bond strength (8.3-11.0 MPa). For the bonding agents that included a phosphate ester system, the shear bond strength showed large scattering depending on the bonding agent (0.2-12.6 MPa). When specimens showed a high shear bond strength, a mixed (adhesive/cohesive) failure mode between bonding agents and amalgam was observed. It was observed that the copal varnish was not as effective as the amalgam bonding agents. The bond strength of the conventional amalgam was similar to that of high-copper amalgam.

Ramos and Perdigao (1997) evaluated the amalgam bond strength to dentin using current dentin adhesives, namely, All-Bond 2 (AB), Amalgambond Plus (AMP), Fuji Duet (FD), One-Step + Resinomer (OSR), OptiBond Dual cure (OPD), OptiBond FL (OPF), Prime and Bond 2.0 + Dyract Cem (PBDC) (Caulk-Dentsply), and Scotchbond Multi-Purpose Plus (SB). Accumulation of the adhesive in the line and point angles of the cavities, and interpenetration between the amalgam and the adhesive, were also observed, except for FD and OPF. PBDC, AMP, and SB provided the highest bond strengths.

When comparing resins of similar chemistry, Ruzickova and others (1997) also found that the formulation with the higher viscosity and filler content gave higher strengths, with Panavia giving particularly good results with both spherical and lathe-cut alloys.

Moisture

In vitro bond strengths of amalgam (Dispersalloy) to etched (37% phosphoric acid) and non-etched enamel have been studied under dry and moist conditions using two bonding agents (Scotchbond Multi-Purpose Plus, All-Bond 2) and a hybrid ionomer. It was found that moisture did not affect the bond strength between amalgam and enamel with the bonding agents used (McRary, Atske & Powers, 1997). In the case of dentin, the results are likely to be different depending on the bonding agents involved. For example, acetone-based bonding agents show higher bond strengths to moist than to dried dentin (Kanca, 1992).

Condensation Techniques

Ratananakin, Denehy, and Vargas (1996) determined the shear bond strength of Tytin amalgam to dentin using All-Bond 2, Amalgambond Plus, Amalgambond Plus with HPA powder, and OptiBond with hand and mechanical condensation. For both condensation techniques, shear bond strengths of Amalgambond Plus with HPA powder were significantly higher that those for OptiBond, while those for Amalgambond Plus and All-Bond 2 were lower. The two condensation techniques did not have a significant effect on shear bond strengths of Tytin amalgam bonded to human dentin. This study is in agreement with a similar study performed by Tjan, Tan, and Berry (1994), with the conclusion that OptiBond performed better than Amalgambond (without HPA) and All-Bond 2 in bonding Tytin amalgam to dentin. It also corresponded to the study by Vargas, Denehy, and Ratananakin (1994), where Amalgambond Plus with HPA performed better than OptiBond, while All-Bond 2 gave lower bond strengths.

Adhesive Liners Effect on Strength

Two *in vitro* studies have demonstrated that bonding with resin adhesive liners affects the mechanical properties of the set amalgam, although with variable results. One study (Charlton, Murchison & Moore, 1991) demonstrated that adhesive resin incorporated into hand-condensed spherical dental amalgam alloy significantly reduced the compressive strength of the alloy, measured at 1 hour, 24 hours, and 7 days. Relatively large standard deviations ranging from approximately 10% to more than 50% of the group

mean for compressive strength were reported, consistent with considerable technique sensitivity. This same study demonstrated the presence of the adhesive resin within the dental amalgam restoration, but did not show its distribution.

A further *in vitro* study (Millstein & Naguib, 1995) demonstrated a significant reduction in diametral tensile strength of dental amalgam condensed into a Teflon-simulated preparation containing an excess of either of two resin adhesives. The amalgam compressive strength was reduced, but not significantly affected by the use of adhesive resin. The authors stated that resin was not restricted to the preparation walls, but there was also partial intermixing of resin within the dental amalgam during condensation.

Another *in vitro* study (Boston, 1997) was conducted to try to determine the distribution of two adhesive liners (Amalgambond Plus and Resinomer with All-Bond 2) within standardized Class I dental amalgam restorations by using thin-section radiography and reflecting microscopy. It was found that those restorations bonded with adhesive liner had greater amounts of non-amalgam substance than did the non-adhesive liner restorations. Both resin liners were capable of being incorporated within the body of a Class I restoration placed with a standard restorative amalgam bonding technique. The pattern of incorporation corresponded to incremental amalgam placement during restoration. Amalgam bonding material was found at the cavosurface margin of the restoration and extended to the occlusal surface of the restoration. This study also proposed that further investigation would be necessary to better understand the pattern of distribution of bonding materials within bonded amalgam restorations, to correlate these patterns with physical properties, and to determine the effects of restorative technique on inclusion patterns and physical properties.

Modes of Failure

Ratananakin and others (1996) examined the experimentally fractured surfaces of amalgam bonded to dentin and revealed that most failures were adhesive, occurring along the dentin-amalgam interface. The adhesive failures occurred at either the dentin-adhesive resin or adhesive resin-amalgam interface. The fracture patterns of all materials tested were also related to their bond strengths. Amalgambond Plus with HPA showed the higher bond strength with the higher percentage of mixed failure.

Ólmez and Ulusu (1995) evaluated the tensile bond strength to deep dentin of primary teeth, for amalgam (3.0 MPa) bonded with Amalgambond Plus with HPA powder (AMP), and compared this to the tensile bond strength of composite resin (6.4 MPa) bonded with AMP. The modes of failure of the specimens were observed under light microscopy and recorded as adhesive, cohesive, or mixed. The amalgam group exhibited an adhesive failure rate of 100%. The modes of failure of the resin composite group were 24% adhesive and 76% mixed. Adhesive failures of resin composite specimens were found only when low tensile strength values were recorded.

In contrast, Eakle, Staninec, and Lacy (1992) examined sections of amalgam bonded to teeth with Panavia resin liner and found that amalgam was intermixed with the resin liner at the interface between alloy and tooth. The mode of failure of bonded amalgam restorations was mixed, but cohesive resin failure predominated.

Another study analyzed the mode of fracture of amalgam bonded to teeth using Panavia 21, All-Bond 2 Primer/Bonding Resin, and Amalgambond Plus (Oliveira, Cochran & Moore, 1996). There was cohesive failure in the amalgam in 35%, 25%, and 15% of the specimens, respectively. This type of fracture is a good indication of effective bonding between tooth and amalgam. The most common type of fracture for all the restored groups was the one occurring at the tooth-restoration interface. The authors concluded that these findings suggested that current bonding procedures could be improved.

Tooth Strength

A restorative material should restore form and function together with the strength of the tooth. Studies have been carried out to evaluate the effect of bonding amalgams to tooth structure on tooth stiffness. One such study conducted by Zidan and Abdel Kereim (1997) compared the stiffness of teeth restored with amalgam, amalgam bonded with Amalgambond Plus with HPA Powder, and composite bonded with Scotchbond Multi-Purpose Plus adhesive. The researchers concluded that restoring the tooth with non-bonded amalgam did not restore lost tooth stiffness. On the other hand, restoring the prepared tooth with bonded amalgam or with bonded composite significantly recovered the lost tooth stiffness.

A similar study by Borchert and Boyer (1996) was aimed at determining whether mesio-occlusodistal (MOD) amalgam restorations placed with resin bonding agents reinforced cusps of molars and if fillers were important. Tytin amalgam was bonded with One-Step + Resinomer, OptiBond dual cure, All-Bond 2, and Amalgambond Plus/HPA adhesive. It was concluded that amalgam MOD restorations bonded with resin adhesives with inorganic fillers reinforced cusps of molars, whereas those without did not.

El-Badrawy (1996) measured the cuspal deflection of maxillary premolars restored with bonded amalgam using strain gauges *in vitro*. The results indicated that bonding amalgam restorations decreased cuspal deflection and consequently assisted in restoring tooth strength.

It has been shown that removal of tooth structure by cavity preparation can weaken teeth and increase their susceptibility to fracture (Larson, Douglas & Geistfeld, 1981). Christensen and others (1991) reported that teeth restored with Amalgambond and amalgam had cusp fracture resistance values almost 50% higher than unprepared teeth.

In contrast, Santos and Meiers (1994), in combining the effects of aging and thermocycling of MOD restorations, did not find amalgams bonded with Amalgambond to be more fracture resistant than conventional amalgam restorations.

Eakle and others (1992) compared the fracture resistance of teeth restored with conventional amalgam with those restored with amalgam bonded to tooth structure. Teeth restored with bonded amalgam fractured at a significantly greater load than did teeth restored with non-bonded amalgam. Bennett (1995) found that amalgam restorations bonded using ED Primer and Panavia 21 had significantly greater resistance to fracture than non-bonded restorations.

Rauvola, Broome, and Simon (1997) measured the resistance to fracture of posterior amalgam restorations replacing one cusp. All teeth were restored with Tytin amalgam utilizing four different retentive features: existing mechanical retention only, one TMS Minim pin (Coltène/Whaledent, Mahwah, NJ 07430), Advance Hybrid Ionomer Cement (Dentsply), or All-Bond 2 adhesive. The study demonstrated that higher resistance was obtained from hybrid ionomer-retained complex amalgam restorations versus those mechanically retained with a pin.

Bailey and Boyer (1997) determined if bonding amalgam to dentin would increase the resistance to fracture of teeth containing a Class I restoration. It was concluded that teeth restored with bonded Class I amalgam restorations were more resistant to fracture than teeth restored with non-bonded amalgam restorations.

Similarly, a study conducted by Oliveira and others (1996) investigated the influence of a number of different amalgam bonding agents on the fracture resistance of teeth containing large mesio-occlusodistal (MOD) restorations. The adhesive systems used included Panavia 21, Amalgambond Plus, Imperva Dual Bond (Shofu, Kyoto, Japan), All-Bond 2 Primer/Bonding Resin, and All-Bond 2 Primer/Liner F. The results demonstrated that the use of the adhesive liners Imperva Dual Bond and All-Bond 2 Primer/Bonding Resin significantly increased the strength of the teeth when compared to a varnish-lined control.

Microleakage

A problem common to dental amalgam restoration (particularly high-copper alloys) is microleakage at the amalgam-tooth interface for a time after placement. Whereas microleakage is primarily investigated *in vitro*, the clinical relevance of these findings may only be seen in relation to the prevalence of sensitivity, and ultimately, secondary caries. Staninec and Holt (1988) compared resin-lined and varnish-lined amalgams using basic fuchsin dye. The results showed that the bonded amalgam leaked less than the varnish-lined restorations.

Kohalmi and Gorzo (1996) conducted an *in vitro* study comparing the marginal adaptation of Class II amalgam and composite restorations. The most important conclusions of the study were (1) in the case of deep Class II restorations, the amalgam had a significantly better adaptation at the gingival margin than the composite restorations, and (2) the adhesive technique reduced but did not eliminate the marginal leakage.

A separate study by Araujo, Rocha, and Garotti (1997) evaluated and compared the effectiveness of two dentin adhesive systems: Amalgambond Plus and Scotchbond Multi-Purpose on marginal sealing at the gingival floor of Class II cavity Dispersalloy amalgam and Z100 composite (3M) restorations. It was found that the use of these two fourth-generation dentin adhesive systems did not completely seal the interface on the gingival floor.

Alloy Factors

Mahler and Adey (1997) evaluated the following alloy factors related to microleakage of amalgam alloys: setting dimensional change, surface roughness, and mix plasticity. It was found that microleakage of amalgams can be reduced by increasing mix plasticity (increase Hg/alloy ratio), decreasing setting contraction, and/or decreasing surface roughness.

Meiers and Turner (1997) evaluated microleakage of dentin bonding agents used with amalgam. They evaluated the long-term effectiveness of certain resin-liner combinations [Amalgambond Plus, Tenure/Panavia, Syntac/DualCem (Ivoclar/Vivadent, Schaan, Liechtenstein), and All-Bond 2/Liner F] in reducing microleakage in Class V cavity preparations restored with Dispersalloy or Tytin and compared those results to previous short-term data. The results indicated that various resin-liner combinations could provide significant protection against microleakage under high-copper amalgam alloys for up to one year. Though corrosion products from high-copper amalgams may eventually seal the interfacial gap between tooth and amalgam restoration, at one year these did not equal the protection provided by three of the four resin adhesive liners.

Several studies have demonstrated that resin-lined amalgam restorations exhibit significantly less microleakage than unlined or varnish-lined restorations (Yu, Wei & Xu, 1987; Staninec & Holt, 1988;

Turner, St Germain & Meiers, 1995). However, like copal varnish, adhesive resins may not be effective in completely eliminating microleakage in all restorations. With time, resin-lined restorations may tend to exhibit a degree of marginal leakage similar to, but certainly no worse than that of varnish-lined restorations. However, unlike copal varnish, adhesive resins appear to maintain their dentinal seal. Saiku, St Germain, and Meiers (1993) have shown that resin-lined amalgams tend to leak at the resin-amalgam interface rather than the resin-enamel or resin-dentin junction.

Recurrent Caries in Vitro

Torii and others (1989) conducted a study in which Panavia EX was used to bond amalgam to tooth structure. Bonded and non-bonded amalgam restorations were incubated in a medium containing sucrose and culture of Streptococcus mutans for five weeks, changing the medium every four days. The results of the study indicated that, in the laboratory, the bonded amalgams showed less penetration of caries along the interface than the non-bonded amalgams, thus showing that an effective seal was present at the amalgam-tooth interface.

CLINICAL STUDIES

Secondary Caries

Recent clinical studies have reported a low incidence of secondary caries around bonded amalgam restorations after two years in clinical service (Belcher & Stewart, 1997), lending support to *in vitro* studies. Staninec and others (1997) retrieved exfoliated deciduous teeth to study clinically-placed bonded and non-bonded amalgam restorations. Caries was found at the margin of one out of three non-bonded amalgams, whereas none of the six bonded amalgams exhibited caries. It must be noted, however, that the incidence of secondary caries in these clinical studies is so low that no meaningful statistically significant conclusions can yet be drawn. In order to test this or any other technique for secondary caries resistance clinically, subjects with high caries rates would have to be used.

Postoperative Sensitivity

In vitro studies have shown that significant reductions in microleakage may be achieved with the use of adhesive resins to bond amalgam to tooth structure. Few studies, however, have addressed the clinical effectiveness of resin liners in reducing sensitivity. Kennington and others (1996) compared short-term postoperative sensitivity in teeth restored with amalgam using a bonded resin liner (3M Scotchbond Multi-Purpose Plus) versus teeth restored using a copal varnish liner. Paired restorations placed by the same operator were evaluated in 20 patients, one lined with adhesive resin and the other lined with copal varnish. Patients filled in a questionnaire on sensitivity on days 1, 3, 7, 14, and 30 postoperatively. It was found that there was no significant difference in postoperative sensitivity between the two-cavity lining materials at any postoperative interval.

Similar studies evaluating postoperative sensitivity have found no significant difference when compared with varnish-lined restorations up to three years (Rosensteil, Rashid & Pagniano, 1996; Browning, Johnson & Gregory, 1997a; Engle & Mahler, 1997). A study evaluating postoperative hypersensitivity between bonded amalgams (with Panavia 21) and non-bonded amalgams one to two weeks after placement, showed no difference between the restorations (Mahler & others, 1996). The study was continued for two more years with the same results (Engle & Mahler, 1997).

Marginal Fracture Studies

Marginal fracture is a characteristic of amalgam restorations in clinical service. The extent of this fracture occurring over a period of time has been used as a measure of clinical performance. It is of significance that marginal fracture is used by dentists as one of the criteria for replacement of restorations, even when there is no definitive evidence of secondary caries (Qvist, Thylstrup & Mjör, 1986).

Some methods of measuring marginal fracture are performed indirectly by using clinically-derived photographs or casts of restorations made during regular sequential patient reviews. These methods include the Mahler scale (Mahler, Engle & Phillips, 1993) and the Index of Marginal Fracture (Fukushima, Setcos & Phillips, 1988). The Mahler scale is made up of photographs selected to cover the entire range of marginal fractures exhibited by many types of alloys, ranging from virtually no fracture to a gross extent of fracture. In studies, clinical photographs may then be compared with the photograph scale in order to assess the extent of marginal fracture. The Index of Marginal Fracture (IMF) is calculated following careful examination and scoring of the extent of marginal defects at restoration margins on casts from clinical impressions made at study recalls. Therefore, it is a quantitative technique for assessing the cavosurface margin of restorations and is expressed as a percentage. Mach and others (1998a) in a clinical study found that there was no significant difference between IMF for bonded and non-bonded amalgam restorations over two years of clinical service.

Clinical Findings

Engle and Mahler (1997) measured postoperative sensitivity and marginal fracture of bonded versus non-bonded restorations at two and three years of clinical service. Marginal fracture evaluation was recorded using a photoevaluation scale. At two- and three-year recall evaluations, there was no statistical difference

found between bonded and non-bonded restorations with respect to sensitivity or marginal fracture.

A three-year clinical study comparing bonded with non-bonded restorations for anatomic form, marginal adaptation, sensitivity, and surface quality was conducted by Mach and others (1998b). Etching, All-Bond 2 Primer A&B, and Bisco C&B resin cement were used for bonding. In addition, 11 unprepared teeth had bonded amalgam sealants placed and evaluated for more than three years. It was concluded that bonded amalgam works as well in traditional preparations as non-bonded amalgam restorations after three years clinically, and bonded amalgam is successful in preparations without undercuts or pits and fissures without preparation.

Staninec and others (1998) examined the efficacy of amalgam also bonded using All-Bond 2 and Liner F into unprepared pits and fissures as a sealant, compared to a conventional resin sealant. At six months, one year, and two years, sealants were examined using modified USPHS criteria. It was concluded from the two-year results that bonded amalgam used to seal unprepared pits and fissures is as effective as resin sealant. Patient acceptance of dental amalgam sealants would be expected to depend on their level of concern for esthetics.

Setcos and others (1998), in a clinical evaluation, found no significant differences in temperature sensitivity, marginal adaptation, anatomic form, or surface roughness between the bonded and non-bonded restorations. However, no restorations bonded by ED Primer with Panavia 21 were lost over two years, whereas three non-bonded restorations were lost from preparations with no deliberate retention. This suggests that bonded amalgams may be found to survive better than non-bonded amalgam restorations in preparations with no deliberate retention. Longer-term reviews may establish this.

A study undertaken by Browning, Johnson, and Gregory (1997b) evaluated the clinical performance of bonded amalgam restorations at 18 months. Anatomic form, marginal adaptation, and secondary caries were evaluated using the criteria of Cvar and Ryge (1971). All restorations were judged to be clinically acceptable at 18 months. Restorations placed with the adhesive liner OptiBond performed as well as restorations placed with conventional bases and liners.

Summitt and others (1998) examined 30 pairs of complex amalgam restorations (replacing one or more cusps) in molar teeth at baseline, six months, one year, and two years. One restoration of each pair was pin-retained and the other retained by Amalgambond Plus (AB+) with HPA powder. Copalite was used in the pin-retained group. There was no sensitivity to cold at two years. Two pin-retained restorations failed, as they required endodontic treatment by year two. The authors concluded that AB+ functioned as well as pin retention at two years.

While many investigations have compared bonded restorations with mechanically-retained restorations, there are also studies that indicate improved performance when the two methods are combined (Imbery, Burgess & Batzer, 1995; Burgess & others, 1997; Uyehara, Overton & Davis, 1998). This is appropriate since so many restorations are replacements, and there are often existing undercuts that may be used in addition to adhesive bonding.

CONCLUSIONS

In addition to providing adequate retention, bonded amalgam restorations may provide distinct advantages over non-bonded amalgam restorations by added tooth reinforcement, decreased postoperative sensitivity, better marginal adaptation, reduced secondary caries, and more conservative preparation.

There is a substantial body of supportive evidence for the advantages of bonding amalgams, a great deal of which is derived from *in vitro* investigations. The degree to which these advantages are realized is proportional to the strength and longevity of the adhesive bond. The few *in vivo* studies available to date show little advantage for bonding in traditional preparations with mechanical undercuts. However, there is evidence accruing that bonded amalgam can be favorably used in other situations, such as large compound restorations, preparations without retention, or as sealants in pits and fissures, notably adjacent to bonded amalgam restorations. Sealing pits and fissures close to a bonded amalgam restoration may be conveniently performed at the same time as the restoration placement.

Despite the apparent advantages of bonding amalgam restorations, for small restorations it is unlikely that bonding will be routinely employed in preference to traditional non-bonded amalgam restorations, nor challenge the ever-increasing use of bonded resin composite. But there seems to be an apparent increased confidence of clinicians in using bonding in the placement of large compound amalgam restorations. Prospective randomized controlled clinical studies over longer periods of time are required to determine the longevity of the bonded amalgam restoration in clinical service. These studies should also cover extended uses for the resin bonding of amalgam, such as for prolonging the life of existing restorations by repairs and additions. Notwithstanding the advantages of the technique, there is an added cost associated with bonding, which should be subjected to cost-benefit analysis. Furthermore, given the increasing applications and use of amalgam bonding, it is suggested that this procedure should be widely included in the pre-doctoral dental curriculum.

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Invited Paper

The Full Gold Crown: An Overview

R V Tucker

For various reasons, the full crown has become the choice of treatment for an increasingly large number of patients needing tooth restoration. Failure rate is high. It has been shown by a statistical study of more than eight million patients that the typical longevity of full gold crowns is between three and four years (Craig 1989). What can be done to correct this and make the full gold crown a more acceptable restoration?

Indications for full coverage have particularly increased because of the increased longevity of patients, restoration of badly-destroyed teeth that in previous years would have been removed, and a high incidence of endodontically-treated teeth. A full crown satisfies the need to bind the tooth together. There are shortcomings, however, which can be partially overcome with proper preparation of the tooth, with concern for detail and tissue health.

PREPARATION

The tooth should be reduced as little as possible, avoiding a cone shape, creating walls that are almost parallel (Figure 1). Paralleling of walls will help avoid unnecessary damage to the tooth, increase retention quality, and allow a more accurate casting due to the thin, vertical walls and lack of excess bulk in the crown. Adequate reduction is necessary on the occlusal plane (Figure 2) to allow enough thickness of gold to prevent wearing through the crown in future years, and to allow for proper contouring of the wax to prevent occlusal interferences.

The margins of a full crown preparation should be almost a knife-edge type margin, rather than a shoulder (Shillingburg, Hobo & Fisher 1974). The rationale for avoiding a shoulder is that the discrepancy in marginal fit of the casting would allow only microns of tooth to be exposed (Figure 3), rather than creating a channel of cement into the tooth at the depth of the shoulder (Figure 4).

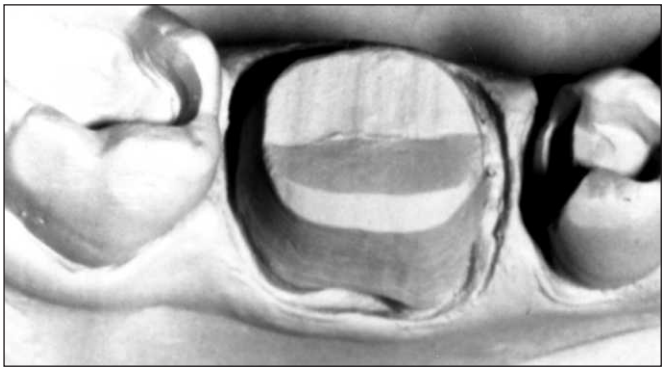

Figure 1. *Minimal reduction is appropriate for gold crowns. Walls should be almost parallel.*

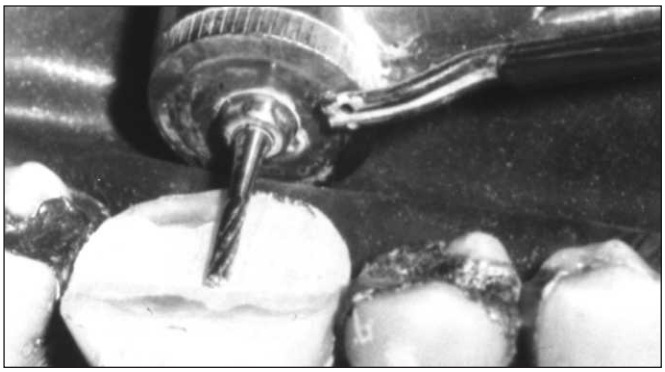

Figure 2. *Occlusal reduction with a straight fissure bur.*

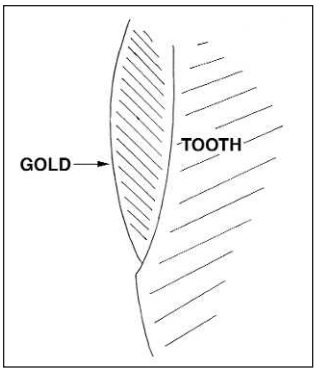

Figure 3. *Diagram suggesting minimal marginal discrepancy with a "knife-edge" margin preparation.*

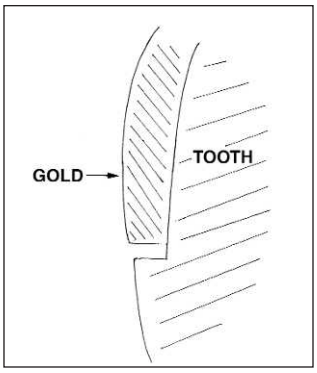

Figure 4. *Diagram suggesting larger marginal discrepancy with a shoulder type preparation.*

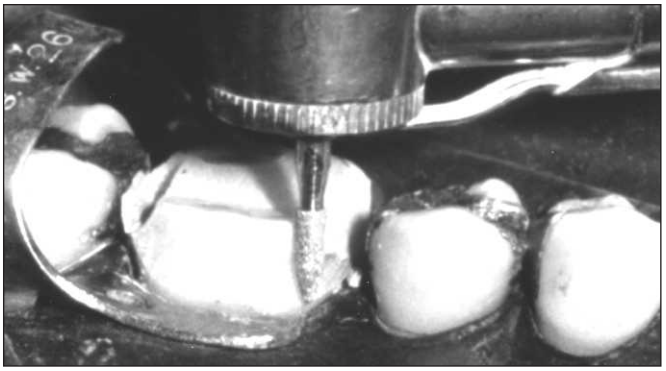

Figure 5. *Reduction of axial walls with the 860-014 diamond.*

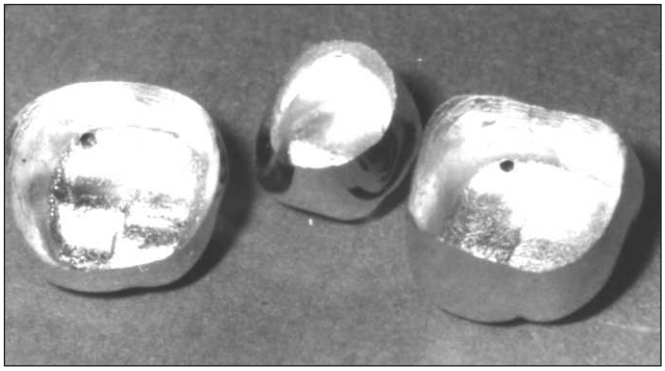

Figure 6. *Castings showing thin axial walls and sharp cervical margin.*

The obvious result of this channel might be marginal caries after the cement has dissolved.

The #860-014 flame-shaped diamond point (Brasseler USA, One Brasseler Blvd, Savannah, GA 31419) accomplishes this. It has a slight curve, which allows reduction of the wall of the tooth without creating a shoulder, but with a clear definition of the margin to allow easy wax-up. The walls are reduced and the margins are painted on the tooth with fine strokes (Figure 5).

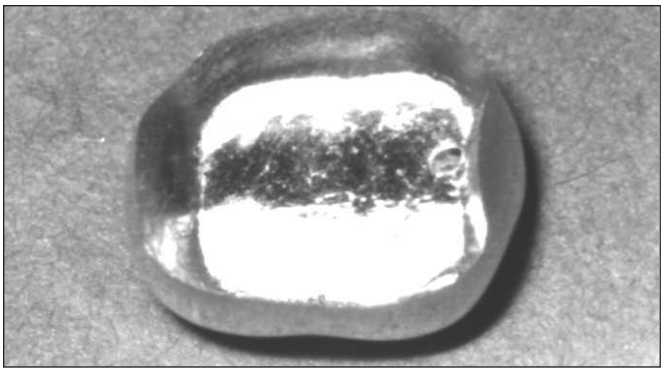

Figure 7. Ca*sting finished and ready to placed.*

Figure 7. *Final restoration in the mouth.*

THE CASTING

Thin walls of the casting are desirable (Figure 6) because they allow increased casting accuracy, and there is a lack of concern for wearing through this nonfunctional area. The thickness of a casting is important because, logically, any error in the laboratory to perfectly compensate for the gold shrinkage as it cools would be magnified by the mass of the gold in the casting. After the casting is made, it must be properly finished. The following laboratory technique satisfactorily accomplishes this.

FINISHING

The pits and fissures should be outlined with a #1 round bur, and polishing should be accomplished with Robertson Brushes (Buffalo Dental Mfg 99 Lafayette, Syosset, NY 11791), first with tripoli (Buffalo Dental), then rouge (Buffalo Dental). The broad surfaces of the cusps and walls of the crown are finished with a series of three types of paper discs (EC Moore Co, 13325 Leonard Street, Dearborn, MI 48126); medium garnet, fine sand, and fine cuttle. The discs, with a slow speed handpiece, allow finishing right to the margin without removing gold from the marginal length. This is accomplished by curving them flat as they rotate over the surface of the crown only to the margin. Final finishing is done with 3/4 inch chamois or felt wheels (Buffalo

Dental), first using tripoli, then rouge to present a casting without scratches or blemish (Figures 7 & 8).

VARIATIONS IN CROWN PREPARATION

Probably the chief disadvantage of full crowns is the adverse periodontal response to placement of the gold margin into the gingival sulcus. It is this author's opinion that tissues are never really healthy again after a crown is placed under the tissue. This is in spite of special care to achieve smooth margins and highly-finished gold with the best contours that we can achieve.

To counter this effect, in concern for the periodontal health of patients, a new and different concept for full crowns was conceived in 1987. After preparing many ivorine dies and extracted teeth, it was determined that a mesial and distal hollow grinding would allow satisfactory retention and resistance form (Tucker 1972). This is done with the buccal and lingual margins outlined above the tissue, perhaps at or near the height of contour. This preserves one of the main purposes of placing a crown—that of binding the tooth together, and maintains the relationship of natural tooth surfaces to the gingival tissue (Tucker 1996).

This preparation is accomplished with the usual #860-014 flame diamond, being careful not to remove the "ears" of the preparation, but hollow grinding the mesial and distal walls (Figure 9). Care should be given so that the axial wall is not leaned into the tooth more than necessary to aid retention. Since this preparation does not go over the bulge of the buccal wall, both buccal and lingual surfaces will not be parallel, and will be short so they would offer little retention.

Adequate retention is achieved by the proximal hollow grinds. Length is achieved interproximally because, commonly, old restorations or lesions require dropping the preparation deep gingivally in this area; and by using the hollow principle, the buccal and lingual "ears" will aid retention and provide buccal and lingual resistance form (Figure 10). It could almost be compared to a MOD onlay with proximal hollow ground walls rather than a proximal box with line angles and point angles. This form still achieves the feature of binding the tooth together, as previously mentioned.

Although it is a full crown, often when seated, it resembles a large onlay and can be finished directly on the tooth. This can result in a rather spectacular restoration, and hundreds of crowns of this design have been placed without reported failure (Figure 11).

Another variation of the full crown, designed in 1989, also allows the margin on the buccal wall to be placed above the tissue. This is for the tooth that does not have a deep lesion on mesial and distal, so that the normal outline would not extend gingivally in this area. The proximal walls would be placed in typical

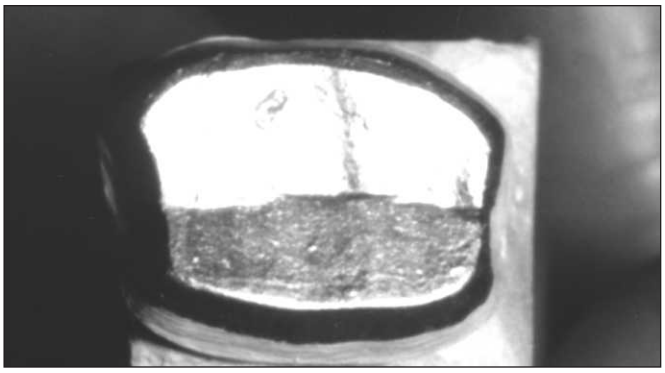

Figure 9. *Die showing hollow ground walls rather than proximal boxes.*

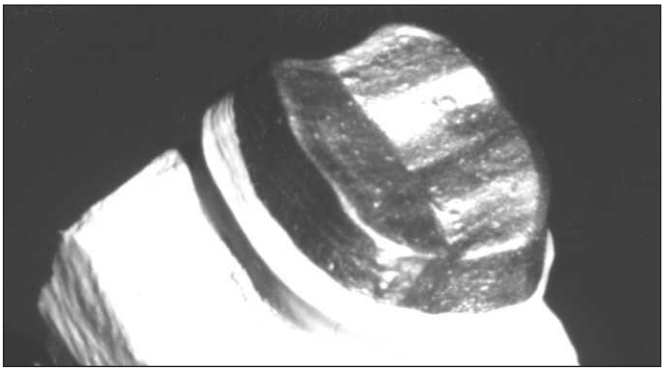

Figure 10. *Another view of the die showing interproximal length of the preparation.*

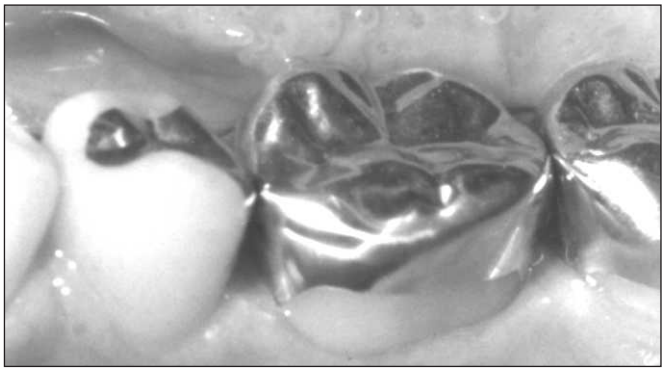

Figure 11. *Final restoration of hollow ground preparation.*

manner, but the buccal wall is prepared with a heavy shoulder that allows it to be parallel to the lingual surface (Figure 12). It provides adequate retention, even though the wall is short, with the margin at the height of buccal contour. This preparation has a butt joint that is easy to finish with the typical procedures of discing and polishing at the time of cementation (Figure 13).

When a crown fits accurately, it is necessary to place an escape-way in it to allow the cement to flow and relieve the hydraulic pressure, which prevents complete seating of the crown. It is also wise to place a die

Figure 12. *Preparation die showing the parallel buccal wall formation.*

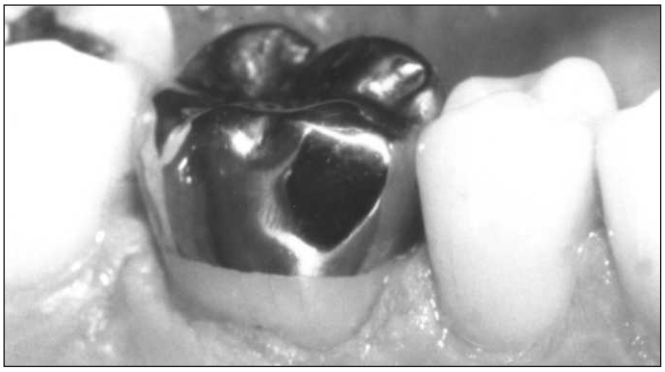

Figure 13. *Finished casting. Note supragingival margin placement.*

relief on the die prior to waxing to assure a more precise fit at the gingival margin.

Full crowns are an excellent restoration when a more conservative type is not indicated, but they, too, require attention to detail if we are to achieve a good clinical result.

Dr Richard V Tucker is in private practice (PO Box 1146, Ferndale, WA 98248). He is the founder of the Affiliated Richard V Tucker Cast Gold Study Clubs, which boast (at last count) 39 active study clubs around the world, with more than 450 members.

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Awards

American Academy of Gold Foil Operators
Clinician of the Year Award

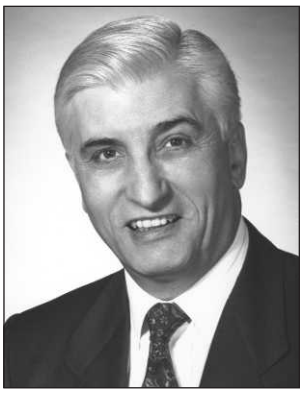
Dr Allan G Osborn

The American Academy of Gold Foil Operators "Clinician of the Year Award" is sponsored by Ivoclar/Williams and is given annually in recognition of the abilities and contributions of one of the academy's members. It is my pleasure to present this award to Dr Allan G Osborn.

Dr Osborn was born in Mount Vernon, New York, but moved (at the age of 3) to the United Kingdom, where he was educated at Repton Preparatory School and Epsom College. He graduated from the University of Sheffield in Dentistry in 1960 and proceeded into internships in both Orthodontics and Oral Surgery. In 1962, he moved to Winnipeg, Canada, and traveled extensively throughout Northern Manitoba and the Arctic by plane and icebreaker, performing dentistry as far north as Grise Fjord on Ellesmere Island.

Allan was introduced to Dr Gerry Stibbs at a two-week Ferrier Course in Edmonton, has been a regular attendee of the annual meetings of the Associated Ferrier Study Clubs since 1972, and a member of the American Academy of Gold Foil Operators since 1975. He founded and mentored the Winnipeg Ferrier Society for 20 years and arranged for, and assisted, both Dr Bruce Smith and Dr Norman Ferguson with two-week instructional courses.

Dr Osborn is a member of the Academy of Operative Dentistry, the American Academy of Restorative Dentistry, the Canadian Academy of Restorative Dentistry and Prosthodontics (for which he has served as both Conference Chairman and Program Chairman), and a Fellow of the International Academy of Dentistry. As a member of the Canadian Dental Association, Allan served on the Council for Accreditation and on the American Association of Dental Schools Executive Committee for Operative Dentistry. He has also been a councilor on the board of the Academy of Operative Dentistry.



Allan G Osborn

Allan has taught as an Honorary Lecturer at the University of Manitoba, assisting Dr George Brass for seven years in cast and direct gold techniques. He has presented table clinics at numerous dental meetings and has been active in continuing education internationally in the areas of direct gold, cast gold, and porcelain inlays. He has authored several articles and a chapter for an Australian medical textbook.

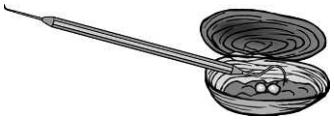
Dr Osborn has worked tirelessly for the Academy of Gold Foil Operators, serving on numerous committees and as president in 1988. He has also been the editor of the Academy newsletter, *The Gold Leaf*, since 1982. Allan has also lectured several times to the Academy and operated at numerous annual meetings. More recently, he has served on the ad hoc Committee for International Meetings and has chaired this committee through four successful European meetings.

Despite all his professional activities and accomplishments, Allan still finds time for numerous hobbies, such as the piano, cooking, photography, his Kawasaki motorcycle, and, of course, travel. He is also devoted to rugby. He served on the Board of the Canadian Rugby Union for 4 1/2 years and, during his playing years, was an outstanding hooker.

In recognition of his service to the Academy and to the dental profession, it is an honor to present the Clinician of the Year Award to Dr Allan Osborn.

David Thorburn

Operative Pearls



Please submit your own clinical tips and techniques to share with your colleagues. Send "pearls" and/or comments on this section via fax (317) 278-4900 or e-mail to editor@jopdent.org.

E-Z GOLD FOR REPAIR OF ABRADED AND ERODED CUSP TIPS

Contributed by:
Dr Lloyd Baum, Loma Linda, CA

Regurgitated gastric acid and other causes often leave cratered enamel walls around an abraded or eroded incisal edge or cusp tip. These craters can best be restored with E-Z Gold (Ivoclar/Williams, 175 Pineview Drive, Amhurst, NY 14228) because, among other advantages, gold and enamel have similar wear characteristics.

Illustrated in Figure 1 is a lesion that could serve as a candidate for E-Z Gold. The concavity of the dentin between the enamel plates needs to be rebuilt to the level of the enamel plates in order to protect the dentin from erosive/abrasive elements.

Following the placement of the rubber dam, a #35 inverted cone but is used to prepare the bulk of the cavity. Figure 2 illustrates a box-like cavity which does not undermine the labial or lingual enamel, but obtains retention at the mesial and distal, where the bulk of dentin is greatest. Cavity depth is 1-1.5 mm. A fissure bur or finishing bur is used to create a labial and lingual level that terminates on the worn surface about 1/3 the thickness of the enamel plate. It should be noted that the worn (abraded) surface of the labial and lingual plates is not included in the preparation, and the operator should NOT attempt to lengthen the incisal edge by building gold over the worn surface.

Figure 1.

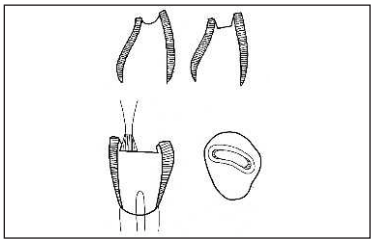


Figure 2.

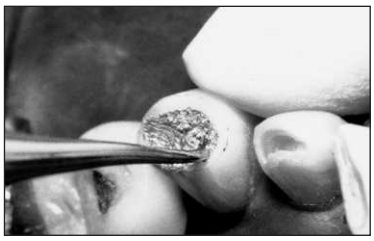


Figure 3.

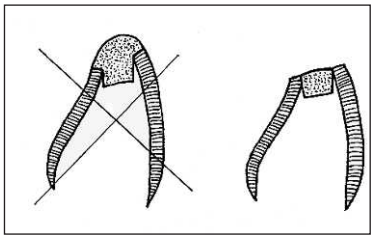


Figure 4.

Figure 5.

Condensation of the gold should be done with small pluggers (Figure 3). The shank of larger condensers may inadvertently pry or fracture away portions of the labial or lingual plates.

It must be remembered that pure gold is softer than casting gold. If an increase in incisal length were needed, a crown would be the restoration of choice (Figure 4). Restoration of eroded lesions with E-Z Gold provides excellent protection against additional incisal wear and/or erosion (Figure 5).

Abstracts

The editor wishes to thank the second-year General Dentistry residents at Wilford Hall United States Air Force Medical Center at Lackland AFB, Texas, for their assistance in the preparation of these abstracts.

Ten-year clinical assessment of three posterior resin composites and two amalgams. Mair, LH (1998) Quintessence International **29(2) 483-490.**

(University of Liverpool, United Kingdom)

This study reports on the clinical performance of three posterior composites and two amalgams after 10 years of service. Thirty Class II restorations each of P-30, Occlusin, Clearfil Posterior (composites), New True

Dentalloy, and Solila Nova (amalgams) were placed. The baseline assessment was made at least one day after and within two weeks of placement. All restorations were assessed for the quality of contact points, gingival bleeding on probing, and the presence of marginal ledges, gaps, or recurrent caries over the course of the study. Amalgams were assessed for the presence of tarnish or corrosion. Resin composites were assessed for the presence of cavosurface marginal staining and general surface staining. For each restoration, a quadrant impression was taken with an addition-reaction silicone-impression material. These were used to make epoxy resin replicas of the restorations for the measurement of wear. The restorations were reviewed after 6 months and 1, 2, 3, 4, 5, and 10 years. At the 10-year review, 61% of the original restorations were available for recall (18 Clearfil, 20 Occlusin, 18 P-30, 15 New true Dentalloy, and 20 Solila Nova). The wear values at 10 years were subjected to a one-way analysis of variance using Scheffe's multiple comparison test (level = .05) to determine significantly different subsets.

Because of the incomplete recall, it was impossible to give an absolute failure rate. There was no evidence of recurrent caries in the restorations examined. The composite restorations were judged to have maintained an adequate color match at 10 years. All but two of the Occlusin restorations had evidence of cavosurface marginal stain, whereas only five of the other composite restorations had evidence of such stain. Throughout the 10 years, there was no evidence of general surface stain except when patients were using chlorhexidine mouth rinses. There was little evidence of tarnish on any of the amalgam restorations, but the amalgams did have signs of surface and cavosurface marginal corrosion. There was no change in the contact point status or gingival condition of any of the restorations over 10 years. Over the 10-year study, one composite restoration was replaced because of excessive wear after three years of service.

Both posterior resin composites and amalgams provided adequate clinical service for 10 years. The author stated that it appears that the bonding layer should be kept as thin as possible to minimize staining. Corrosion was not found to be a major problem in well-condensed amalgam restorations. Wear resistance does not appear to be attributed purely to the filler particle configuration, and the commonly held view that "more small particles" will confer greater wear resistance, needs further consideration. The wear rate of all the materials decreased with time (especially after five years). The results of this study indicate that, provided that the material is handled proficiently, posterior resin composites can provide relatively long-term restorations. The choice of material should be dictated by factors such as the size and position of the cavity (as well as the patient's desires and economics) rather than by arguments about biocompatibility.

Conclusions

Three posterior composites and two amalgams placed in 1985 in a dental school environment, provided adequate clinical service for 10 years. However, the operator factor is likely to be very important, and this study does not indicate how the materials would perform under less favorable clinical placement conditions.

Volumetric microleakage assessment of glass-ionomer-resin composite hybrid materials. deMagalhães CS, Serra MC and Rodrigues AL (1999) Quintessence International **30(2) 117-121.**

(School of Dentistry, Federal University of Minas Gerais, Belu Horizonte, Brazil)

Microleakage in Class V cavity restorations with margins in dentin is still a concern when using adhesive techniques. Glass-ionomer cements have many favorable characteristics, including adhesion to tooth structure, fluoride release, coefficient of thermal expansion similar to dentin, and low setting shrinkage. Weaknesses of glass-ionomer cements include lack of fracture toughness, susceptibility to moisture and dehydration, and technique sensitivity. Hybrid materials have been introduced in an attempt to improve the physical properties and clinical handling characteristics of glass-ionomer cements. These are referred to as either polyacid-modified resin composites or resin-modified glass-ionomer cements.

This study sought to quantify the marginal leakage of three glass-ionomer-resin composite hybrid materials and compare the results with those of a glass-ionomer cement and a bonded resin composite system. A total of 105 extracted human teeth were cleaned and stored in distilled water at room temperature to prevent dehydration. A standardized Class V cavity preparation—1.5 mm deep and 1.5 mm in diameter—was placed on the facial root surface approximately 4.0 mm below the cementoenamel junction. The teeth were randomly assigned to five groups of 21 teeth each and restored with the following materials: Ketac-Fil aplicap glass-ionomer cement; Z-100 resin composite and ScotchBond Multi-Purpose Plus dentin adhesive system; Vitremer resin-modified glass-ionomer cement; Photac-Fil Aplicap resin-modified glass-ionomer cement; and Dyract polyacid-modified resin composite.

The restorative materials were placed in one increment. They were stored in distilled water at 37°C for 24 hours and wet-finished with the Sof-Lex graded series of aluminum oxide disks. After 24 hours in distilled 37°C water, the specimens were thermocycled for

500 cycles in water between 5°C and 55°C with a dwell time of 60 seconds in each bath. After cycling, the specimens were placed in 2% methylene blue dye at room temperature for 24 hours.

To determine the quantitative amount of microleakage, root sections 6x6x4 mm, including the restorations, were removed, ground into powder, and individually immersed in glass tubes containing from 0 to 10 μg dye/ml. Root sections were restored with each tested material in the same way that the specimens were. After the roots were dissolved (48 hours), the solutions were centrifuged and absorption was determined using a Beckman DU-65 spectrophotometer. Marginal leakage was recorded as μg/ml.

Data were analyzed by nonparametric Friedman's test. The sum of ranks of marginal leakage showed no statistically significant differences at the 95% confidence level among the five restorative systems evaluated. The volumetric microleakage performances of the materials tested were similar.

Conclusions

Factors other than marginal microleakage, including physical properties, esthetics, ease of manipulation, cariostatic effect, cost, and operator preferences should be considered when a restorative system is selected for cervical restorations.

Long-term durability of dentin bonds made with a self-etching primer, in vivo**. Sano H, Yoshikawa T, Pereira PN, Kanemura N, Morigami M, Taemi J and Pashley, DH (1999)** Journal of Dental Research **78(4) 906-911**

(Hokkaido University School of Dentistry, Department of Operative Dentistry, Sapporo, Japan)

Longevity of adhesive bonds is still one of the areas of current interest in adhesive dentistry. Several studies have demonstrated a dramatic decrease in bond strength after five-year storage in water. The use of monkey teeth *in vivo* provides a useful screening model between *in vitro* tests and human clinical trials. Therefore, the purpose of this study was to evaluate the durability of resin bond strength to dentin restored in the oral environment of a monkey under occlusal stress, as well as to test the hypothesis that the adhesive interface would show morphological changes *in vivo* over time.

Shallow saucer-shaped dentin cavities (3 mm wide, 4 mm long, and 1-1.5 mm deep) were prepared in 12 intact teeth of one monkey under general anesthesia. The teeth were restored at three different times; immediately, and 180 and 360 days before the teeth were extracted for study. The cut enamel and dentin surface were treated with a self-etching primer (Clearfil Liner Bond II) for 30 sec, then air-dried. A thin layer of adhesive resin (Clearfil Liner Bond II) was applied on the primed surface and light-cured for 20 sec. A layer of resin composite (Clearfil Photo Posterior) was then placed on the cavity and light-cured for 60 sec.

One day after the monkey was sacrificed, specimens of the three time periods were subjected to the microtensile bond test at a crosshead speed of 1 mm/min. The surfaces of the failed bonds were observed under a filled emission scanning electron microscope (FE-SEM). Bond strength measurements were successfully performed and were stable at approximately 19 MPa during the one-year testing. SEM observations of the failed surfaces revealed that porosity increased over time at the top of the hybrid layer and within the adhesive resin. Degradation of resin composite appeared to occur at the junction of the filler particles and the resin matrix.

Conclusions

Long-term bonds can be assessed *in vivo* by the combined evaluation of the microtensile bond strength and SEM morphological examination of the adhesive interface.

Durability of tunnel restorations in general practice: a three-year multi-center study. Pilebro CE, van Dijken JWV, and Stenberg R (1999) Acta Odontologica Scandanavia **57(1) 35-39**

(Institute of Oral Biology and Public Dental Health Service, Ulmea University, Sweden)

Class II tunnel preparations are used on proximal carious lesions which require minimally invasive treatment, thereby preserving the intact marginal ridge and avoiding potential damage to the proximal surface of adjacent teeth. Currently, the application of a glass ionomer in the proximal surfaces is advocated for its cariostatic influence on the treated tooth, as well as on the adjacent proximal surface. The aim of this *in vivo* multi-center study was to evaluate over a three-year period the durability of tunnel restorations performed by a group of general practitioners.

Twelve dentists familiar with the tunnel restorations placed the restorations over one year. The carious lesions were classified into two groups—D2 or D3. The D2 lesions showed a U- or V-shaped radiolucency, which reached or penetrated the DEJ but did not spread in the dentin. The D3 lesions displayed obvious radiolucency in the dentin. The access for the tunnel preparation was made through the occlusal fossa just inside the marginal ridge. A matrix was placed, etched with 10% polyacrylic acid, and Ketac-Silver was used

for the initial fill, and then the preparation was re-etched with 37% phosphoric acid and filled with hybrid composite. Partial tunnels were defined as restorations which did not reach the outer proximal surface. In all other cases, the restorations were defined as total tunnels. A total of 374 tunnel restorations were performed in 272 patients. Molars received 212 restorations (57%), and 162 were placed in premolars (43%). Baseline bitewing x-rays could only be evaluated in 228 cases, the rest of the radiographs could not be used due to poor quality or missing radiographs. The baseline radiographs assessed filling defects, such as air bubbles or proximal overhangs. The quality of the restoration was evaluated annually up to three years. Clinical evaluation included the presence of marginal ridge, fracture, dissolution of the filling material, postoperative sensitivity, and new caries.

Immediate baseline bitewing radiographs revealed that 14% showed defects or failures. Nineteen restorations (8%) showed radiographic signs of non-excavated dentinal caries within the newly-restored teeth. At the one-year recall, the most common failure was due to marginal ridge fracture (3.5%). No restoration was replaced because of caries during the first year. After two years, another 31 restorations were replaced with a higher incidence of enamel and dentin caries. At the three-year recall, 18 restorations were replaced due to marginal ridge fracture. For the duration of this study, a total of 61 of the 305 (20%) restorations were replaced. The number of restorations showing untreated progressive dentinal caries increased during the study. After three years, almost half (83 out of 175) of the enamel lesions showed progression from baseline and/or since the previous evaluation. In total, 41% of the restorations showed either untreated dentin caries or enamel caries with progression. This study showed that the number of failures in tunnel preparation is higher than earlier reports. The use of Ketac-Silver with a low concentration of leachable fluoride may explain why the progression of caries was not prevented. The authors recommend consideration of topical fluoride treatments in conjunction with tunnel preparation. The use of bitewing x-rays to identify carious lesions was found to be low in sensitivity, especially for smaller lesions.

Conclusions

In comparison to posterior composite or amalgam, the rate of replacement for tunnel restorations was much higher. The tunnel preparation should not be regarded as being a general solution for the operative treatment of proximal caries. High rates of marginal ridge fracture and the inability to remove caries due to poor visual access should lead clinicians to limit this treatment modality to small dentin lesions and exclude patients with high caries risk.

Nd:YAG laser effects on the occlusal surface of premolars. Myaki SI, Watanabe IS, Eduardo CdP, Issao M (1998) American Journal of Dentistry **11(3):103-105.**

(Departmento de Anatomia, Universidad de Sao Paulo, Brazil)

When a high-power laser irradiates an enamel surface, depending on the time of exposure, type of active medium, or energy density, the dental enamel may melt and change morphology after recrystallization. Among high-power lasers, Nd:YAG is the most widely used in dentistry. In this study, the effects of pulsed Nd:YAG laser irradiation on the occlusal surface of human premolar teeth was evaluated *in vitro*.

Twenty human premolars, extracted for orthodontic reasons, were stored in saline solution at 4C and randomly divided into two experimental groups (n=10). Before the laser irradiation, the occlusal fissure was painted with black ink in order to increase the absorption of the laser beam by the enamel surface. Group 1 was exposed to the full extension of the occlusal fissure. The irradiation was 2.0 watts, with a repetition rate of 20 Hz and 124.3 J/cm energy density for three minutes. In Group 2, the occlusal fissure was divided into halves, and one-half lased and one-half untreated to serve as the control. After irradiation, teeth from Group 1 were fractured longitudinally to observe the deepest portion of the fissures. The morphologic changes in Group 2 were noted, and both groups were sputter-coated and observed with scanning electron microscopy.

In Group 1, some fissures were sealed by the melting and recrystallization of the enamel. The enamel also exhibited a highly-roughened surface containing different types of trabeculae. The SEM images revealed that the deep fissures were not sealed, or that the fissure was covered by the superficial melted and recrystallized enamel. The bottom of the deep fissures were not sealed and exhibited spaces at the base. The teeth in Group 2 showed superficial sealing of the lased fissures. The non-irradiated surface was unaffected and maintained original morphology. Enamel rugosities caused by the melting/recrystallization were numerous. In the adjacent enamel, different sizes of crater formation were noted. At high magnification, the presence of granules and vitrified areas was seen.

Work by Horowitz has shown that fluoride does not satisfactorily reach and protect pits and fissures, suggesting a need for an alternate method to combat caries in these areas. The present study showed that pulsed Nd:YAG laser irradiation in the output defined can cause melting of the enamel surface and, in some

cases, sealing of the fissure after recrystallization. Sealing of the fissures did not occur uniformly, with shape and depth being important limiting factors. Shallow fissures allowed the laser beam to reach the bottom of the fissure and resulted in sealing. In cases of deep and/or narrow fissures, the beam did not reach the bottom and space was left.

Conclusions

Application of a fluoride solution after laser treatment may improve resistance to demineralization, and this combination may be of importance in preventing pit and fissure caries.

Coronal leakage: Endotoxin penetration from mixed bacterial communities through obturated, post-prepared root canals. Alves J, Walton R, and Drake D (1998) Journal of Endodontics **24(9) 587-591.**

(University of Iowa College of Dentistry, Dows Institute for Dental Research, Iowa City, IA)

Bacteria may penetrate root canal obturating materials, influencing periapical tissues. Such leakage is one of the major causes of endodontic failure. Endotoxin, a potent inflammatory agent, may penetrate faster than bacteria. The purposes of this *in vitro* study were to assess the penetration of post-prepared root canals by endotoxin emanating from mixed bacterial communities, and compare and contrast the penetration rate of bacterial endotoxin with bacterial cells.

The mixed bacterial communities were prepared with four anaerobic species present in necrotic pulps. Experimental teeth were freshly extracted, non-contaminated human maxillary incisors, and canines. Keeping the apical foramen patent, laterally condensed gutta-percha with sealer obturation and post-space preparation was accomplished. The model to measure penetration of substances consisted of an upper chamber, which contained an opening for the tooth crown and received the bacterial suspension every few days, simulating coronal leakage. Root surfaces were sealed to restrict leakage to the apical foramen. The root of the tooth extended into a lower chamber containing a solution which was removed weekly and assayed for endotoxin and bacteria. A negative control group had its root surfaces (including the apices) sealed. A positive control group was obturated with a single cone and no sealer. The specific times and amount of leakage for endotoxin and bacteria were measured and compared.

Results indicated that apical leakage of endotoxin, a potent irritant, does occur. Bacteria also penetrated the obturating materials, but more slowly than the endotoxin. Positive controls exhibited leakage through the apex in 15 days. Endotoxin leakage occurred in as quickly as eight days, with an average of 23 days. The mean bacterial leakage was 62 days.

Conclusions

These findings support the need for adequate obturation using a sealer. They also indicate that penetration of these potentially damaging substances can occur rapidly, leading to a periapical response and possible treatment failure. The need for an immediate and proper coronal restoration after root canal treatment is reinforced.

Comparative radiopacity of flowable resin composites. Murchison DF, Charlton DG, Moore WS (1999) Quintessence International **30(3) 179-84.**

(General Dentistry Residency, Wilford Hall USAF Medical Center, Lackland Air Force Base, TX)

Modern resin composites are manufactured to exhibit a radiodensity that is distinctive, yet similar to tooth structure. Flowable composites have recently been introduced into the marketplace and have had clinical acceptance due to their ease of placement. These low viscosity composites are not as highly filled as formulations for universal use. Manufacturer's product information sheets list filler types as glass and claim radiopacity, yet no studies have evaluated the radiopacity of these materials. The purpose of this study was to determine the comparative radiopacity of eight newly-introduced flowable composite resins and compare their radiodensity to that of enamel, dentin, and a widely-used composite marketed for universal application. The study evaluated nine experimental groups (eight flowable composites and a universal hybrid resin composite). Enamel, dentin, and aluminum standards were also evaluated.

Forty-five composite specimens were fabricated in cylindrical molds and exposed to a curing light for 120 seconds. All specimens were stored dry until testing. Specimens were placed on a size 2 dental radiograph film, along with an enamel and dentin disk and a 4-mm thick amalgam cylinder. Radiographs were standardized. The photographic densities of the radiographs, along with the background film of all experimental groups, were measured with a transmission densitometer (X-Rite). An aluminum step wedge was radiographed and used as an internal standard for establishing aluminum equivalent values. A computer-assisted densitometric image analysis station (CADIA) was used as a concurrent method of analysis. Five determinations of radiodensity were made, with the mean values and standard deviations calculated. Data was analyzed using a one-way ANOVA and post-hoc Scheffe's multiple comparison test.

Only three of the flowable composites exhibited radiopacity equal or greater than that of enamel. The radiopacity of the others was not statistically significantly greater than that of dentin.

Conclusions

The flowable composites Tetric Flow, Flow-It LF, and Crystal-Essence had radiopacity equal to or greater than the aluminum equivalent and enamel. Densitometry and computer-assisted digital analysis indicated that five of the eight flowable composites failed to equal or exceed radiopacity values for the aluminum standard or enamel. A high correlation was found between transmission, densitometry, and digital pixel values, both methods of analysis seem appropriate for comparative radiodensity studies.

Effect of Different Liner Treatments on Postoperative Sensitivity of Amalgam Restorations. Gordan VV, Mjör IA, Hucke RD, Smith GE (1999) Quintessence International **30 (1): 55-59.**

(University of Florida School of Dentistry, Gainesville, FL)

Tooth sensitivity may be a problem after placement of amalgam restorations. Bases and liners have been widely used to eliminate or reduce postoperative dentinal sensitivity. Different liners and bases used have included copal varnishes, dentinal bonding agents, and glass ionomer liners, or the use of no liner at all. The purpose of this clinical trial was to assess the sensitivity experienced by patients following Class I and Class II amalgam preparations following different treatments of the prepared cavities.

Four groups (n=19) were compared. The groups were as follows: Group 1—no liner; Group 2—two coats of Copalite (Cooley and Cooley); Group 3—a dentin adhesive resin liner, Scotchbond Multipurpose (3M); and Group 4—modified glass-ionomer liner Fuji Bond LC (GC). All teeth with a history of sensitivity, or patients who were taking analgesics that could alter the pain perception, were eliminated from the study. A rubber dam was utilized for all procedures, and the amalgam used was a dispersed-phase amalgam (Original D, Wykle). Patients were then followed-up via phone on days two and seven. If there was still sensitivity at day seven, those patients were then followed-up on days 14, 30, and 90.

All groups experienced some sensitivity by day two, and the depth of the restorations did not seem to affect the results. At seven days, none of the restorations lined with the Fuji Bond LC had any sensitivity, and only half of those without a liner had sensitivity. On day 14, none of the no-liner group had sensitivity, but in the Scotchbond Multipurpose (17%) and Copalite (22%) groups, the sensitivity experienced on day seven remained. On day 30, the sensitivity was reduced in all but one tooth restored with Copalite, and two teeth restored with Scotchbond Multipurpose. All of these restorations had pulpal floor extensions into the middle third of the dentin. There was no residual sensitivity by day 90.

Conclusions

The Fuji Bond LC and no-dentin treatment had fewer than one-third of the teeth with short-term sensitivity and none lasted until day 14. Almost one-third of the teeth restored with Scotchbond Multipurpose or Copalite experienced short-term sensitivity that in isolated cases lasted for up to 30 days. At day 30, sensitivity was nonexistent in most teeth, and after 90 days, all sensitivity had subsided irrespective of the dentin treatment provided.

Direct-placement gallium restorative alloy: A 3-year clinical evaluation. Osborne JW, Summitt JB (1999) Quintessence International **30(1):49-53.**

(University of Texas Health Science Center, San Antonio, and University of Colorado, School of Dentistry)

Gallium-containing direct restorative material has been a topic of discussion for approximately 70 years. The most recent interest surfaced during the early 1990s with another call for a mercury-free alternative to conventional amalgam alloy. A gallium restorative material, Galloy, has received ADA approval, however, several clinical trials have shown that Galloy results in a high percentage of postoperative sensitivity, a high rate of tarnish, fracture of restorations and teeth, and a high rate of pulpal necrosis. The purpose of this report is to assess the three-year clinical results of Galloy as a direct restorative material.

Nine patients were utilized for the study, each in need of 2-4 Class I restorations. One operator placed 30 direct-placement gallium alloy restorations following proper isolation and tooth preparation. Fifteen of the cavity preparations received an unfilled triethylene glycol dimethacrylate, urethane dimethacrylate, EBADMA, and bis-GMA-containing resin sealer (SDI). The gallium alloy was then placed and the resin was again applied to completely seal all alloy surfaces. The other 15 restorations received Amalgambond in the same manner as the previous group. This procedure was done to ensure total encapsulation of the gallium alloy and freedom from moisture contamination at the

time of placement. Photographs were taken of all restorations at baseline, three months, six months, one year, two years, and three years and were used to assess restorations for tarnish and for fracture through the body and at the margins. The restorations were evaluated clinically to detect tooth fracture and roughness of the alloy surface and to assess tooth sensitivity by the patient.

The results show that after three years, 32% of available restorations were severely tarnished and 37% exhibited a rough surface. There also appeared to be a disproportionate number of tooth fractures as a result of Galloy placement. There was no difference between the restorations lined with Amalgambond and those lined with SDI resin.

Conclusions

This study suggests that Galloy, used with either of two sealing resins to prevent fluid contamination, is not a suitable restorative material.

Book Reviews

Hybridization of Dental Hard Tissues

Nobuo Nakabayashi
David H Pashley

Published by Quintessence Publishing Co, Inc, Chicago, 1998. 136 pages, 320 illustrations, $48.00, softbound.

This is an excellent publication which draws together our current knowledge of resin-based adhesive systems. The first author (Nakabayashi) was responsible for the early research that developed the concept of hybridization, and is therefore in a unique position to review the literature. Pashley's contribution to dentin hybridization stems from his earlier work on dentin permeability. However, a significant omission is any information about the authors and their credentials, other than affiliations and addresses. Their very important contribution to dentin bonding and dentin physiology has not been identified.

There are chapters on Evolution of dentin-resin bonding, Properties of dentin, Acid conditioning and hybridization of substrates, Characterization of the hybrid layer, Quality of hybridized dentin, and Clinical applications of hybrid layer formation. Each chapter is clearly written and is accompanied by excellent diagrams, photographs, and scanning electron micrographs. There are around 400 references, including some from 1998. It is, therefore, an invaluable resource for further work in the area. Although there are several review papers on resin-dentin bonding, this is the only comprehensive text available.

The first chapter takes the reader through adhesive mechanisms of conventional cements and explains the concepts of resin hybridization. There is a useful section on definitions, which is essential in order to fully understand the rest of the book. The authors then expand on the hybrid layer and on early dentin bonding mechanisms. Some of the impediments to successful bonding are discussed, including the smear layer.

Chapter 2 (Properties of dentin) discusses the mechanical properties which influence adhesion, and also the interaction between dentin and acrylate monomers that influence the diffusion process.

Chapter 3 examines in detail the preparation of the dentin surface for bonding and the subsequent placement of primer and adhesive. All the options of technique and materials are clearly explained with good diagrams.

The hybrid layer is considered in detail in Chapter 6. Various experimental techniques are explained, including surface analytical methods and scanning electron microscopy.

Chapter 7 examines the quality of the bond over time and the concepts of microleakage and nanoleakage. The latter is a relatively new concept which is becoming increasingly important in bond longevity.

The final chapter describes the clinical applications of resin-dentin bonding, including biocompatability issues and direct pulp capping.

The book is essential reading for all those carrying out laboratory and clinical research on adhesion of resin-based materials to enamel and dentin. It is probably too detailed for the general practitioner to read throughout, as much of the material is research oriented, but would be a very useful resource for information on specific issues.

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The Complete Denture: A Clinical Pathway

I Michael MacEntee

Published by Quintessence Publishing Co, Inc, Chicago, 1998. 136 pages, 320 illustrations, $48.00, spiral bound.

The author, professor, and chair of the division of Prosthodontics, Department of Oral Health Sciences at the University of British Columbia, indicates that he has devoted much of his academic career to the condition of edentulism. Contributors who are faculty in his department have written three of the 12 chapters. The author has offered this book primarily to the clinician inexperienced in complete dentures, but also to the mature dentist and the undergraduate dental student.

The text describes a single technique of complete denture fabrication, which the author indicates is based on scientific principles derived from empirical research and clinical experience reported over the last 100 years or so. Detailed descriptions of alternative techniques are avoided in order to focus on one pathway to a complex task. While this is not intended to be a definitive reference on complete denture prosthodontics, the author has achieved his objective in this textbook by providing an organized, concise, well-written, well-illustrated description of a single technique in the management of the complete denture patient. It utilizes numerous high-quality, black and white, clinical and laboratory photographs to support the text, with many of the pages containing more illustrations than text. The information in each chapter is supported by a sufficient number of references which can be used as a source of additional information should the reader desire.

Useful features of the book are the two appendixes. The first is a list of instrumentation required and the procedures to be accomplished at each appointment. The second is an information sheet on living with new dentures, which can be given to the patient. The book is well-indexed, allowing the reader to quickly find answers to clinical questions.

I would recommend this book for the dentist with limited experience in complete denture treatment, and for the dentist who requires a review prior to treating the occasional complete denture patient.

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Creighton - General Dentistry

Creighton University, a Catholic, Jesuit institution, is initiating a search for the position of assistant/associate professor in the newly-formed Department of General Dentistry. The department is part of a reorganization that will encompass the previous disciplines of Comprehensive Dental Care, Operative Dentistry, and Oral Diagnosis and Radiology. Responsibilities include teaching at the preclinical and clinical levels, didactic instruction, research, and participation in continuing education activities. Qualifications include a DDS/DMD degree from an accredited US dental school and post-graduate training is desired. Licensure or eligibility for licensure in Nebraska is required. Opportunity for intramural or extramural practice is available. Send letter of interest and curriculum vitae with a list of three references to: Dr Mark A Latta, Chair, General Dentistry Search Committee, Creighton University School of Dentistry, 2500 California Plaza, Omaha, NE 68178. Creighton University is an Equal Opportunity/Affirmative Action Employer.

University of Maryland-Dental School-Restorative

The Department of Restorative Dentistry announces a permanent full-time (1.0 FTE) Assistant/Associate Professor, tenure-track position. Responsibilities include teaching primarily operative dentistry and/or biomaterials in the predoctoral program and conducting research. A DDS/DMD or equivalent degree is required, and research experience and/or additional training/education beyond the dental degree in a relevant biomedical field is recommended. The candidate must be eligible for licensure in Maryland. Prior teaching experience is desirable. The successful candidate will be expected to conduct independent research, and as such, preference will be given to those individuals with evidence of prior research activity and the poten-

tial to successfully compete for external research funds. The University of Maryland, Baltimore, is an AA/EEO/ADA employer and has a strong commitment to the principle of diversity in all areas. Salary and rank are commensurate with qualifications and experience. Intramural practice is available and encouraged. Please submit a letter of interest, a curriculum vitae, and names and addresses of three references to: Dr Morton Wood, Chairman, Department of Restorative Dentistry, Dental School, University of Maryland, 666 West Baltimore Street, Baltimore, MD 21201, (410) 706-1841. For best consideration, applications should be submitted no later than April 1, 2000.

University of Iowa – Chair, Department of Operative Dentistry

The University of Iowa's College of Dentistry is presently conducting a search to fill a full-time tenure-track faculty position as Chair of the Department of Operative Dentistry at the rank of Associate or Full Professor. Major responsibilities include: providing strong departmental leadership, faculty development and mentoring, and maintaining intra-collegiate and university collaborations. The position will be available immediately; screening will begin immediately. Applicants must have a DDS/DMD degree or equivalent, advanced education training in operative dentistry or related field, a record of scholarship; evidence of effective teaching, and relevant administrative experience. Academic rank and salary will be commensurate with qualifications and experience. Submit CV and three letters of recommendation to: Search Chair, Dr Georgia Johnson, Department of Periodontics, College of Dentistry, University of Iowa, Iowa City, IA 52242. The University of Iowa is an affirmative action/equal opportunity employer; women and minorities are encouraged to apply.

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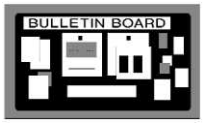
This program provides dentists and related postdoctoral dental health care professionals with advanced skills and knowledge in selected aspects of infectious diseases and their control, and experience in design and management of related research. The candidate selected for the Fellowship will be accepted into the program starting July 1, 2000 and will receive funding for two years through a stipend. The Fellow is expected to participate in ongoing and independently-developed formal research as well as the didactic and clinical education programs in infectious disease control at the dental school. Overall, the Fellow will define an individual program of study and research, with advice and consultation of the program director. Contact Dr Robert Cooley, Fellowship Director, University of Texas Health Science Center at San Antonio, 7703 Floyd Curl Drive, San Antonio, TX 78284-7914. Phone: (210) 567-3450, fax: (210) 567-3443, e-mail: cooley@uthscsa.edu.

Announcements



American Academy of Gold Foil Operators Annual Meeting

1-4 November, 2000
Honolulu, Hawaii

It's not too early to begin planning for your Hawaiian Luau! A one-half day clinical session and two half-day essay sessions are planned. Headquarters hotel will be the fabulous JW Marriott Ihilani Resort and Spa.

For more information, contact: Dr Ronald Harris, AAGFO Secretary-Treasurer, 17922 Tallgrass Court, Noblesville, IN 46060, phone: 317-867-0414, fax: 317-867-3011, or e-mail: piperon@earthlink.net.

Tucker Institute Clinical Course

A clinical course in conservative gold castings, mentored by Dr Richard V Tucker, will be held June 12-16, 2000 at the University of Washington Dental School. For course information, please contact Dr Dennis Miya at 206-244-1618; fax 206-431-9800.

To Our Contributors and Readers

Operative Dentistry has been extremely fortunate in establishing a reputation for quality that has resulted in increased manuscript submissions each year. We have seen our popularity grow to the point where we received 110 articles for review in 1999. However, while this is an outstanding achievement for our journal, we have developed a backlog of accepted articles that has put us in a position of being 18+ months from acceptance of a manuscript to its publication. This is an unacceptable length of time in our rapidly-changing profession. It is too long for authors to wait for their work to appear in print, and too long for our readers to wait for timely scientific information. Therefore, we will be expanding *Operative Dentistry* from 72 to 104 pages for the remainder of 2000. This should allow us to "catch up" and eliminate our backlog of accepted papers. Our subscribers should note that this is being done at no increase in cost to them, thanks to our new Corporate

Sponsorship program that provides the necessary funds to cover the additional printing and mailing costs. If our submission rate continues to rise, we will probably continue to publish a larger journal. Our hope is that you will share the news of the expanded issues, encourage your colleagues to subscribe, as well as encourage their research submissions to *Operative Dentistry*. Our editorial team's goal is that once a manuscript has been accepted, it will be published within 10 months.

Michael A Cochran, Editor

Operative Dentistry Home Page

We hope all our readers will take advantage of the information available by accessing our Internet home page. Our address is: **http://www.jopdent.org/**

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